NATIONAL RENEWABLE ENERGY LABORATORY
GOLDEN, COLORADO

SUBCONTRACT ACO-9-29067-01
PROCESS DESIGN AND COST ESTIMATE OF CRITICAL EQUIPMENT IN THE BIOMASS TO ETHANOL PROCESS

REPORT 99-10600/14
LIQUID/SOLID SEPARATION

REVISION 1
MARCH 6, 2001
WEB

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REPORT 99-10600/14

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REPORT 99-10600/14

LIQUID/SOLID SEPARATION

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1. EXECUTIVE SUMMARY

The National Renewable Energy Laboratory (NREL) under the direction of the Office of Fuels Development at the U.S. Department of Energy has, over the years, developed a process for converting cellulosic biomass to fuel ethanol based on NREL and subcontracted research and standard engineering practices. Three specific variations of the process were considered for the work in this subcontract: Pretreatment with Enzymatic Hydrolysis (P100), Two-Stage Countercurrent Acid Hydrolysis (P200), and Two-Stage Dilute Acid Hydrolysis (P300).

In Process 100, biomass feedstock in chip form is introduced to the plant and screened. The chips are passed to a scalper screen to remove very large materials and then onto a chip thickness sizing screen. We assume that approximately 20% of the incoming material will be oversized and will require processing through a singe disc refiner system. The disc refiner reduces oversized material to less than 19 mm, suitable for the pretreatment reactors. The biomass is pretreated to make it more susceptible to acid penetration. During pretreatment, much of the hemicellulosic portion of the biomass is hydrolyzed into soluble sugars in a continuous hydrolysis reactor. This reactor uses steam and dilute sulfuric acid to initiate hydrolysis. Afterwards, the liquid portion of the pretreated slurry must be separated from the solids to facilitate conditioning of the liquid portion to remove compounds, such as acetic acid, that may be toxic to downstream fermentative organisms. Once the liquid stream is conditioned properly (most likely via ion exchange and overliming), it is recombined with the solids and sent to fermentation.

This process uses simultaneous saccharification and co-fermentation (SSCF) to hydrolyze cellulose and ferment the resulting glucose and other sugars present to ethanol in the same vessel. As this design currently stands, a portion of the pretreated hydrolysate is drawn off and used to produce cellulase enzyme. The enzyme is then added to the fermentation vessels. A recombinant ethanologen is used to ferment multiple sugars to ethanol. The resulting beer is then sent to distillation and dehydration to purify and concentrate the ethanol. The lignin portion of the original biomass gets carried through the system and exits with the distillation bottoms. This lignin is used as fuel for the burner/boiler system in the plant. As a result, it must be dewatered sufficiently to achieve proper combustion.

Process 200 uses no acid in the first stage and a countercurrent reactor design in the second stage of hydrolysis to convert the hemicellulose and a large portion of the cellulose in the biomass to soluble sugars. The second-stage reactor separates the solids and liquor containing dissolved sugars. The solids are sent to the boiler and the liquid is sent to the oligomeric reactor and flash tank. The liquor is then neutralized and sent to fermentation. The back end of the process is the same as Process 100, but the process stream is liquid only.

Processes 200 and 300 differ from P100 in that no enzymes are used. All hydrolysis is accomplished thermochemically. / In the first stage of hydrolysis, the hemicellulosic portion of the biomass is

hydrolyzed to soluble sugars. These sugars must then be washed from the slurry prior to the second stage of hydrolysis or else they will be degraded at the more severe conditions. The soluble sugar stream is neutralized (with stoichiometric amounts of lime) and sent to fermentation. The solids stream, primarily cellulose and lignin, is sent to the second stage of hydrolysis to further hydrolyze cellulose to glucose. After hydrolysis this stream is also sent to fermentation. The back end of the process is the same as Process 100.

Separation of solids from liquid streams and washing of the separated solids are required in the P100 and P300 processes. Liquid/solid separation is also required after distillation in the P100 process. NREL contracted with Harris Group Inc. (HGI) to do an interactive study with NREL engineers to identify liquid/solid separation equipment best suited to achieve the process goals, facilitate testing of that equipment, and develop associated costs for equipment alternatives that satisfy those goals.

The basis for the design and equipment sizing is a biomass feed rate of 2000 dry metric tons per day. Testing was done utilizing hardwood chip feedstock. Other feedstocks, including corn stover, sugar cane bagasse, softwood chips, and rice straw, will probably be commercialized before hardwood chips.

1.1 Post-Distillate Liquid/Solid Separation

~ 42%

The process objectives for liquid/solid separation equipment for P100 post-distillate slurry are to minimize moisture in the solids as well as minimize solids in the separated liquid. Centrifuges at Baker Hughes and Alfa Laval were tested. A Pneumapress pressure filter was also tested. The pressure filter produced cake solids of 38% by weight with total liquid solids of 2.97%, while the centrifuges produced cake between 20% and 26% solids with total liquid solids of 4.27% by weight. Based on budgetary equipment quotations, the estimated installed- cost for an Alfa Laval centrifuge system is $8,800,000. The estimated installed cost for the Pneumapress system is $8,100,000 with an estimated average power demand of 830 kW and an estimated annual power cost of approximately $332,000. The Pneumapress pressure filter is recommended as the best equipment for this application based on testing and equipment evaluation done to date.

1.2 Process P100 Ambient Pressure Liquid/Solid Separation

The process objective for liquid/solid separation for Process P100 is to minimize acetic acid carryover in the solids while limiting the wash water to 132,000 kg/hr (equals 0.58 lb water/lb feed based on 230,545 kg total feed to liquid/solid separation). An acetic acid level of 3.3 g/kg in solids was established as the required maximum with 1.65 g/kg as the desired level. Two horizontal belt filters, a pressure filter, and a filter press were tested.

The proposed Black Clawson horizontal belt filter would limit acetic acid carryover in solids to 1.7 g/kg with 300,000 kg/hr (1.29 lb water/lb feed) of wash water and an estimated installed cost of $27,000,000. The projected wash water flow significantly exceeds the 132,000 kg/hr limit. Black Clawson utilized test results to project the number of wash stages required to achieve the required acetic removal efficiency.

The proposed Baker Hughes horizontal belt filter used two stages of countercurrent washing to meet the targets for acetic acid in the final cake. Baker Hughes has not provided data defining the specific amount of acetic carryover in solids. The amount of wash water required is 1144 gpm, which is 15% above the maximum wash filtrate (989 gpm) allowed. The estimated installed cost of this option is $6,300,000.

The Pneumapress pressure filter produced a residual acetic acid level of 0.9 g/kg, which is below both the required and desired acetic acid levels. This acetic acid residual was achieved with 0.58 lb of wash water per lb of feed (or 133,000 kg/hr of wash water). The wash water flow is also very close to the wash water maximum required. The estimated installed cost of a Pneumapress pressure filter is $8,500,000 with estimated average power demand of 980 kW with an annual electrical power cost of $392,000.

The Pneumapress pressure filter is recommended as the best equipment for this application based on testing and equipment evaluation done to date.

1.3 Process P300 Ambient Pressure Liquid/Solid Separation

The process objective for liquid/solid separation for the P300 process is to maximize sugar recovery from the solids with 95% removal solids required and 98% removal desired. Two horizontal belt filters, a pressure filter, and a filter press were tested.

The proposed Black Clawson horizontal belt filter with six wash stages would remove 95% of the sugar in the solids. The estimated installed cost of the equipment is $49,700,000. Black Clawson utilized test results to project the number of wash stages required to achieve the required sugar removal efficiency.

The proposed Baker Hughes horizontal belt filter, with two stages of countercurrent washing, removed 95% of the sugar in the solids. This would require 1144 gpm of wash water, which is slightly above the maximum wash filtrate (1065 gpm) allowed. To increase the sugar recovery to 98% would require three stages of washing and 2204 gpm of wash filtrate. The estimated installed cost of this option is $6,300,000.

The proposed Pneumapress pressure filter would produce 97% sugar recovery. The estimated installed cost for a system with Pneumapress pressure filters is $8,500,000 with estimated annual electrical power cost of $392,000.

The Pneumapress pressure filter is recommended as the best equipment for this application based on testing and equipment evaluation done to date.

1.4 Process P100 Elevated Temperature Liquid/Solid Separation

Testing of a Pneumapress bench scale unit took place at NREL's facilities in Golden, Colorado. No quantitative analyses were done on resultant filtrate or cake solids from these tests. However, from qualitative observation, the results appeared to be promising. NREL has plans to perform some general liquid/solid separation on a pilot scale Pneumapress filter. If those tests are successful and if funds are available, a pilot scale unit would be purchased. This unit would be capable of operating at elevated temperature and pressure conditions.

2. OBJECTIVES

The key objective of this work is to improve the process design and accuracy of the estimate for segments of the process requiring liquid/solid separation. NREL engineers will incorporate the results of this study into the NREL model with the assistance of HGI. The design estimate is for the "*Nth*" plant to be built in order to eliminate costs associated with a "one-of-kind" or first system built.

3. DISCUSSION

Liquid/solid separation is required in the following process locations:

- After distillation in Process P100
- After first-stage hydrolysis reactor elevated temperature and pressure, Processes P100 and P300
- After first-stage hydrolysis reactor ambient pressures, Processes P100 and P300

All equipment considered for these process applications is current technology, commercially available, and in use in other industries in similar liquid/solid separation process functions. Report 10600/2, Liquid/Solid Separation Vendor Comparison, outlines the basis for equipment and vendor selection and can be found in Appendix F.

The following sections discuss process objectives; equipment options; test results; equipment-specific information including size, operating principles, and expected performance based on test data; as well as power requirements and the capital costs associated with each equipment option.

3.1 Liquid/Solid Separation Equipment

A range of liquid/solid separation equipment was bench scale tested for the various process applications. Following is a brief description of each equipment type.

3.1.1 Centrifuge

The decanter-type centrifuge use centrifugal force to accelerate the sedimentation of solid particles to be separated from a liquid. The suspension to be treated is fed into a rotor composed of a bowl and screw. These turn at high speed, the screw slightly faster than the bowl. The screw conveys and evacuates the decanted solids toward the conical end of the bowl while the clarified liquid is evacuated from the opposite end. Decanter centrifuges provide continuous (as opposed to batch) processing of slurry mixtures.

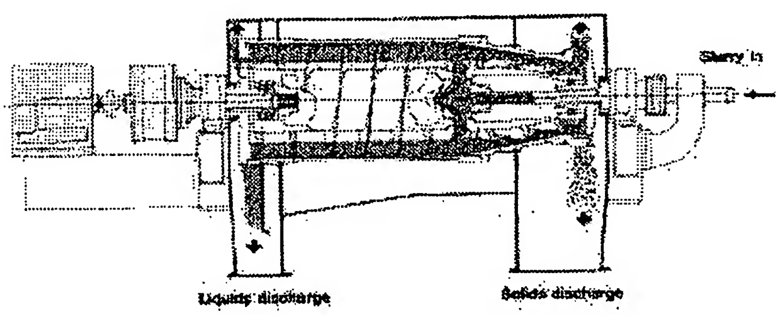


Figure 1.Centrifuge (Alfa Laval)

3.1.2 Filter Press

Filter presses (also called plate and frame presses) operate in a batch mode. First, a pneumatically controlled hydraulic pump applies high pressure to securely close the plates and seal them against internal bypass and/or excessive external leakage. As the illustration indicates, wet slurry is then pumped in through the

center inlet and forced into chambers, which are formed by the vertically oriented matching recessed plates. The pumping action serves as the motive force to provide liquid/solid separation.

Vendors of this equipment offer various enhancements including diaphragm expression, heated water diaphragm expression, vacuum evaporation of liquid from the cake. Cake washing can be accomplished by introducing wash water to the cake after dewatering. Each plate has a filtrate drainage area that is covered with a cloth filter media that traps particles. As the solids build up, they act as filter medium, allowing only clear filtrate to pass through for discharge through the outlet ports. As the chambers fill with cake, the differential pressure increases to the maximum design limit and the stream of filtrate reduces. The plates are opened at the end of a filtration cycle and the cake is discharged.

Filter presses are used in a wide range of applications including minerals, pharmaceuticals, municipal sludge, and gypsum. Liquid/solid separation performances of filter presses vary significantly with the characteristics of the solids. In general, inorganic materials such as gypsum can be dewatered to higher degree than organic material. Size ranges of machines can vary from 2 ft^2 of filter area to over 10,000 ft^2 of filter area. Filter presses can often obtain dryer cake solids than other types of liquid/solid separation equipment. However, they are batch operation, can have large space requirements, and tend to be somewhat more expensive than alternative technologies. Because there is no positive mechanism for removing cake from filter presses, cake release characteristics should be verified for use of this equipment. It is possible to wash cake solids in a filter press by adding a wash cycle to the operation. Because the cake is oriented vertically, "short circuiting" of wash liquid can occur.

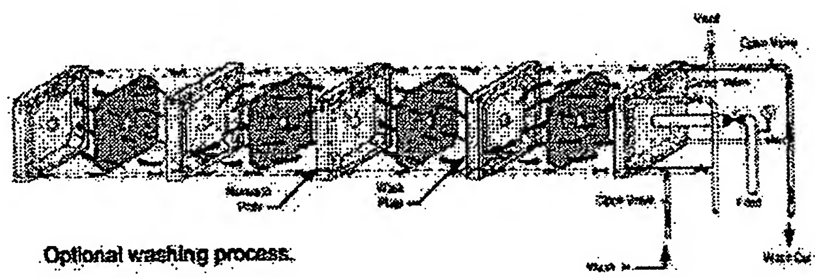


Figure 2. Filter Press (generic)

3.1.3 Belt Filter Press

A belt filter press is a dewatering device that applies mechanical pressure to slurry. The slurry is sandwiched between two tensioned belts by passing those belts through a serpentine of decreasing diameter rolls. The machine can actually be divided into three zones: gravity zone, where free draining water is drained by gravity through a porous belt; wedge zone, where the solids are prepared for pressure application; and pressure zone, where medium, then high, pressure is applied to the conditioned solids.

Typically, a belt filter press receives a slurry ranging from 1% to 4% feed solids and produces a final product of 12% to 50% cake solids. Performance depends on the nature of the solids being processed. Belt presses are commercially

available in widths up to 3 meters. They are used in a wide range of industries including pulp and paper, municipal sewage sludge, and minerals processing. Advantages include continuous operation as opposed to batch and relatively low initial cost. Filter belts can be a significant operating cost, particularly where abrasive solids are being dewatered. Belt presses tend to obtain lower cake solids than other dewatering methods including filter presses, pressure filters, and in some cases centrifuges. Because of their "open" design, hooding and ventilation systems are required to contain fumes, odor, and moisture.

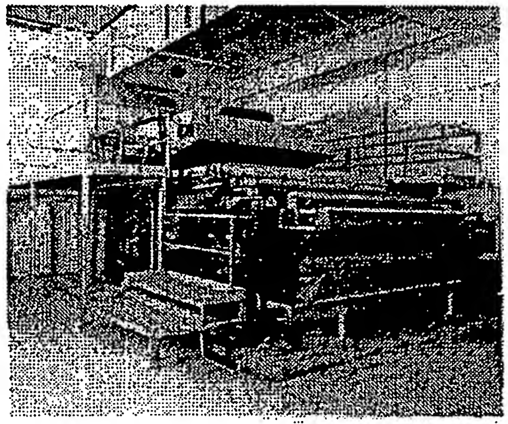


Figure 3. Belt Press (generic)

3.1.4 Horizontal Belt Filter

Horizontal belt filters operate in a continuous mode. These machines are best applied where washing of solids is required. Dewatering is accomplished at each wash stage with vacuum applied to the cake through the filter media from below. Both Thermo Black Clawson and Baker Hughes make horizontal belt filters. While construction details of these machines differ significantly, both use similar principles of operation. Feed is introduced to a filter belt ("wire" – see note below). Liquid is extracted through the filter belt, while solids are retained on the belt. Shower water is applied to solids to remove sugar and/or acetic acid utilizing multiple-stage countercurrent washing. Solids are discharged onto a conveyor. These machines can provide high washing efficiency with a relatively low wash-water-to-solids ratio. Liquid-to-solids wash ratios vary significantly, depending on the characteristics of the solids.

Note: Wire is a term used in the pulp and paper industry for the fabric media used to support and dewater incoming slurries. Fabric composition is typically synthetic fiber such as polypropylene that is suitable for the chemistry and temperature of the application.

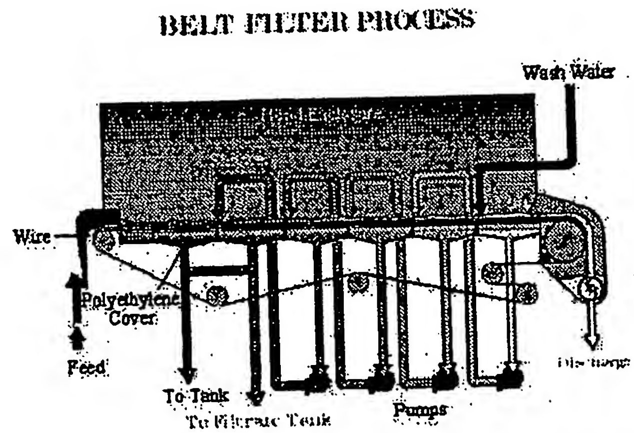


Figure 4. Horizontal Belt Filter (Thermo Black Clawson)

3.1.5 Pneumapress Pressure Filter

A Pneumapress pressure filter operates in a batch mode. Slurry is pumped into cavities formed by multiple horizontally oriented plates. Air pressure applied to solids captured on filter media drives liquid from the solids cake. The pressure filter provides batch liquid/solid separation as follows:

(1) Slurry is pumped into filter chambers formed by lowering the upper plate onto the filter media. Filtrate is collected at the lower plate and flows out through the filter outlet.

(2) Compressed air or gas forces the liquid from the solids retained on the filter media and dries the solid "cake." The cake may be washed after initial liquid/solid separation.

(3) The upper plate raises and the filter media indexes forward, discharging the cake cycle to the operation.

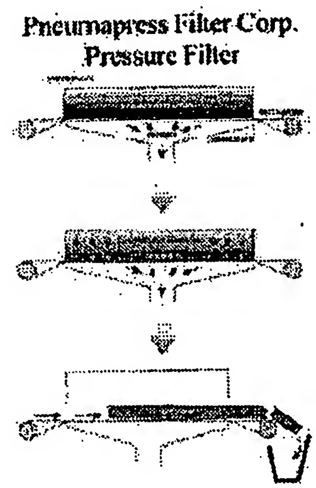


Figure 5. Pressure Filter (Pneumapress Press Filter Corp.)

Because the cake is oriented horizontally, efficient displacement washing is possible. Pressure filters are utilized in a wide range of applications and industries including gypsum, food, pharmaceuticals, and power. The Pneumapress pressure filter is capable of obtaining very high cake solids, depending on solids characteristics. It can also be utilized for high (greater than atmospheric) temperatures and pressures.

3.1.6 Extractor

Crown Iron Works makes an "extractor" that utilizes countercurrent wash flow to extract soluble components of feedstock. A sample of the hydrolyzate slurry was sent to Crown Iron Works for evaluation. The feed slurry has a solids concentration of about 28%. Crown stated the maximum discharge solids concentration that could be expected from its machine would be 15% to 18%. Hence, without additional liquid/solid separation equipment, this equipment would not function in this application. Based on this information, the extractor was not considered to be viable technology for this application.

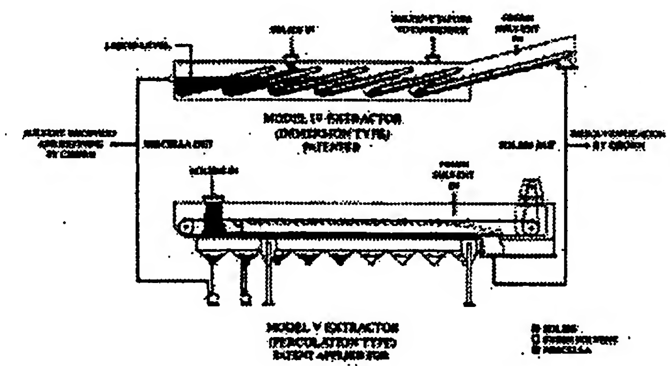


Figure 6. Crown Iron Works Extractor

3.2 Liquid/Solid Separation After Distillation

Liquid/solid separation of the bottoms after the first-effect evaporator is required in the P100 process. The solid fraction generally consists of lignin and unreacted cellulose, while the liquid fraction is primarily water. Process objectives include the following:

- Production of solids with minimum moisture content – The solids are used as a fuel in a burner. Waste stack heat will be utilized to evaporate the remaining water in the solids. One of the process objectives is to minimize the amount of boiler heat used in drying solids after liquid/solid separation.
- Production of filtrate with minimum solids – This water is fed back to fermentation. A minimal amount of solids can be accommodated in the fermentation process.

3.2.1 Materials of Construction

This is a moderate- to low-corrosion application. Sulfuric acid concentration in the feed slurry is approximately 0.1%. Standard industrial corrosion-resistant metallurgy such as Type 316 stainless steel is appropriate for wetted components

of metal process equipment where temperatures are maintained below approximately 92°C at this acid concentration. The feed stream temperature is 86°C for this process step.

3.2.2 Test Material

Test material for post-distillate liquid/solid testing was prepared as described in the NREL report entitled *Experimental Plan EPD0002 – 100L SSF of Pretreated Yellow Poplar* and can be found in Appendix F.

3.2.3 Equipment/Vendor Options

A range of equipment is available for this application. Centrifuges are currently shown on NREL's process flow diagram. The options that were tested include centrifuges, filter presses, belt presses, and pneumatic pressure filters. Three vendors were selected to test equipment to determine the optimal technology to achieve the process goal. Report No. 10600/2, Liquid-Solid Separation (Appendix F) outlines the vendor selection basis. The following vendors and equipment options were tested for this application:

Manufacturer	Equipment
Alfa Laval Separation	Centrifuge
Baker Hughes	Centrifuge, filter press, belt press
Pneumapress Filter Corporation	Pressure filter

3.2.4 Test Results

A summary of test results for each of the vendor tests is provided below. Test reports, data, and an NREL sample analysis for each of the tests may be found in the applicable vendor appendix.

3.2.4.1 Centrifuge

Alfa Laval and Baker Hughes performed a bench scale "spin tests" of the post-distillate slurry. Spin tests generally provide an indication of feasibility of liquid/solid separation of slurry. The solids concentration obtained in a full-size machine is generally better than can be achieved in a laboratory test. (See Table 1.)

3.2.4.2 Filter Press

Baker Hughes performed laboratory bench scale liquid/solid separation tests with post-distillate slurry for its filter press. The results are summarized in Table 2. NREL analysis of samples from these tests may be found in Appendix B.

Table 1
Centrifuge Spin Tests

Item	Unit	Value	
		Alfa Laval	Baker Hughes
Feed slurry			
Total solids	% by weight	7.26	no data
Total suspended solids	% by volume	23.	no data
Viscosity	cP	13.5	no data
Test conditions			
Spin time	minutes	1.5	no data
Centrifugal force	G's	1,500	no data
Slurry temperature	°C	86	no data
Separated streams			
Cake total solids	% by weight	19.9 [1]	22.9 [2]
Cake description		Firm	Heavy mud consistency
Liquid total solids	% by weight	4.26 [1]	3.07 [2]
Liquid dissolved solids	% by weight	3.54 [1]	2.83 [3]

[1] Alfa Laval test data
[2] NREL analysis of Baker Hughes samples, average value of three tests
[3] Baker Hughes test data

Table 2
Baker Hughes Filter Press

Item	Unit	Value
Feed slurry		
Total solids	% by weight	no data
Total suspended solids	% by volume	no data
Viscosity	cP	no data
Test conditions		no data
Separated streams		
Cake total solids (Option A)	% by weight	range 34.9 – 39.7 [1]
Cake total solids (Option B)	% by weight	range 39.7 – 44.4 [1]
Liquid total solids	% by weight	2.67 [2]
Liquid dissolved solids	% by weight	no data

[1] Baker Hughes test data
[2] Results of a single test

3.2.4.3 Belt Filter Press

Baker Hughes did bench scale liquid/solid separation testing of a belt press. The Baker Hughes report excludes any data or discussion of results. The belt press data in Table 3 is from an NREL analysis of samples from belt press testing of post-distillate slurry that Baker Hughes did. It is notable that polymer was utilized to dewater these solids on the belt press. See Baker Hughes' report dated 2/5/01 (Appendix B) for additional test results.

3.2.4.4 Pressure Filter

Pneumapress Filter Corporation did bench scale liquid/solid testing of the pressure filter at its facility in Richmond, California (Table 4). Cake solids and filtering time are both significantly affected by cake thickness. Cake thickness is controlled by the amount of slurry introduced per unit area of filter. Hence, equipment sizing is directly affected by this cake thickness.

3.2.4.5 Test Comparison Summary

Table 5 provides a summary of average percent solids by weight for cake and filtrate/centrate products from testing.

3.2.5 Equipment Costs

Below are capital costs for the Pneumapress and centrifuge options. Capital cost information will be provided for belt presses and filter presses when it becomes available from Baker Hughes. Pneumapresses are the recommended technology. Hence, a more detailed capital cost estimate has been developed for this equipment. All estimates exclude the costs of buildings. Electrical energy costs are provided for a Pneumapress installation.

3.2.5.1 Centrifuge

Alfa Laval states that the laboratory scale tests for the centrifuge do not provide sufficient information on definitive sizing of equipment for the process flows. However, it does provide a good indication of the viability of centrifuges for the application, the type of centrifuge to apply to the process, and a general idea of the level of liquid/solid separation that can be accomplished with centrifuge technology. Based on the results of the tests, Alfa Laval estimates that between five and eight P7600 centrifuges would be required. Alfa Laval suggested that using seven machines as a basis for a capital cost estimate would provide an appropriately conservative estimating approach. The price does not include auxiliary equipment such as pumps, tanks, and conveyors. HGI estimates an installed cost for this system to be $8,800,000. A preliminary equipment list is included in Appendix A.

Table 3
Baker Hughes Belt Press Tests

Test	Item	Unit	Value
D-1	Cake solids	% by weight	27.32
D-2	Cake solids	% by weight	26.90
D-2F	Cake solids	% by weight	34.16
D-3	Cake solids	% by weight	24.74
D-4	Cake solids	% by weight	25.33
D-4E	Cake solids	% by weight	28.39
	Cake solids	avg % by weight	27.81
D-1	Gravity drain liquor	% by weight	2.48
D-2	Gravity drain liquor	% by weight	2.35
D-4	Gravity drain liquor	% by weight	2.40
	Gravity drain liquor	avg % by weight	2.41

Table 4
Pneumapress Pressure Filter

Item	Unit		
Test run		D1	D2
Slurry introduced to pressure filter	ml	240	100
Air pressure	psig	100	100
Slurry temperature	°C	80 to 86	80 to 86
Time for filtrate to clear	seconds	120	20
Blowdown time	seconds	60	30
Cake thickness	in.	½	5/32
Filtrate quality	--	Clear	Clear
Cake total solids	% by weight	41.96	88.04
Cake description		firm w/wet surface	very firm w/dry surface
Liquid total solids	% by weight	2.87	2.95
Filter area	in^2	3.14	

Table 5
Percent Total Solids by Weight

	Alfa Laval		Baker Hughes		Pneumapress	
	Cake	Liquid	Cake	Liquid	Cake	Liquid
Centrifuge	19.9%	4.26%	22.09%	3.07%	---	---
Belt press			27.81%	2.41%		
Filter press			34.9%–39.7% [2] 39.7%–44.4% [3]	2.67%		
Pressure filter					88.04% [1]	2.95%
Feed slurry	% by wt	7.26%	Measured by Alfa Laval (See Appendix A)			

[1] Based on 5/32-in. cake
[2] Option A
[3] Option B

3.2.5.2 Pressure Filter

Pneumapress provided a budgetary quotation for this application. Four Pneumapress pressure filters would be required for the configuration that provided 88% cake solids during testing. An equipment list and cost estimate may be found in Appendix D. The estimated installed cost for a liquid/ solid separation system utilizing Pneumapress pressure filters is $8,100,000. Cycle times were considered in sizing equipment.

For an application with 50% of the current design capacity (flow rate to the pressure filter in kg/hr), two of the four pressure filters would be eliminated. Hence, the total estimated cost of a liquid/solid separation for 50% of current design capacity is $4,400,000.

For an application with 150% of the current design capacity, six pressure filters would be required. It is estimated that the total installed cost for liquid/solid separation for a plant with 150% of the original design capacity cost would be $11,613,000.

The estimated installed horsepower for a Pneumapress installation is 1490 hp with an estimated average power demand of 830 kW. Assuming $0.05/kW-hr and 8000 hours of operation per year, the annual electrical energy operating cost is estimated to be $332,000. Horsepower for this equipment can be scaled linearly with feed rate to the equipment.

3.2.5.3 Filter Press

Baker Hughes provided budgetary quotations for two equipment options. The first (Option A) was for five Model 2000FBM-103-PP-RP-HS-225-32 mm filter presses complete with accessories and controls. This option is a non-membrane filter press that is expected to achieve cake solids of between 37.3% and 39.7%. Estimated installed equipment cost for this system is $17,300,000.

Option B is for five Model 2000FBM-106-PP-mem/RP-HS-225-32 mm with accessories and controls. This option is a membrane-type filter press that is expected to achieve cake solids of between 39.7% and 44.4%. Estimated installed equipment cost for this system is $24,500,000.

3.3 Liquid/Solid Separation First-Stage Hydrolysis, Ambient Pressure/Temperature, Processes P100 and P300

Liquid/solid separation is required after the first-stage hydrolysis reactor in the P100 and P300 processes. The solid fraction consists of unreacted solids. The process objectives of this liquid/solid separation are as follows:

- **Process P100** – Remove the maximum amount of toxins from the solids as practical. Toxins consist of acetic acid and other soluble fermentation toxins. The amount of wash water required to accomplish this should be limited to a maximum of 132,000 kg/hr

(0.58 lb water/lb feed) because this is the total amount of water added to fermentation. The required level of residual acetic acid in 3.3 g/kg in solids with a desired level of 1.65 g/kg.

- **Process P300** – Remove soluble sugar from the solids. Sugar that carries forward to the second-stage hydrolysis will be destroyed. The desired level of sugar recovery from the feed slurry is 95% with a desired level of 98%.

3.3.1 Test Material

First-stage hydrolyzate slurry utilized for these tests was generated from pulp-size aspen hardwood chips at the TVA facility in Muscle Shoals, Alabama. The test material was made in a zirconium-lined digester with a liquid/solids ratio of 4:1 and an acid concentration of 0.55% in the liquid phase. The test was conducted at a temperature of 173°C and a pressure of 112 psig with a retention time of 15 minutes.

TVA collected slurries from first-stage hydrolysis and analyzed them for sugar content, acetic acid, HMF, furfural, and moisture content. The average moisture content was 59.7% and 59.3% for each of two samples tested. The results of the analysis for the remainder of the parameters are given in Table 6.

Table 6
Composition of First-Stage Hydrolyzate Slurry from Pilot Plant in mg/ml

	Drum 11	Drum 13	Drum 14	Drum 16	Av
Glucose	9.90	11.60	11.60	10.70	10.95
Xylose	76.20	62.90	80.00	82.00	75.3
Galact.	<1.25	<1.25	—	<1.25	<1
Arab.	2.25	1.90	1.90	2.25	2.0[illegible]
Mann.	11.70	8.40	9.33	11.70	10.75
Acetic acid	21.10	18.00	19.80	21.20	20.03
HMF	0.29	0.36	0.31	0.31	0.32
Furfural	1.60	1.78	1.74	1.60	1.32

Note: The description of how material was produced and these results were extracted from TVA's April 14, 1999 report, Acid Hydrolysis Support, First And Second Stage Hydrolysis Testing, MPO No. DCO-8-18081-0 (Appendix F).

3.3.2 Materials of Construction

The P100 and P300 liquid/solid separation functions are moderate corrosion applications. Sulfuric acid concentration in the feed slurry is approximately 0.4%. Standard industrial corrosion-resistant metallurgy such as Type 316 stainless steel is appropriate for wetted components of metal process equipment where temperatures are maintained below approximately 82°C at this acid concentration. Because both the P100 and P300 filtrate streams are processed below this temperature downstream of liquid/solid separation, the process stream temperature can be dropped prior to liquid/solid separation to allow use of 316 stainless steel.

3.3.3 Equipment/Vendor Options

Because the primary goal for both the P100 and P300 processes is solids washing with a limited amount of water, displacement countercurrent washing is a desirable approach to get the best washing results with limited wash water. Equipment types were limited to those that could do countercurrent washing. The equipment options tested for this application include horizontal belt filter, pressure filter, and filter presses. Screw presses, belt presses, and decanter centrifuges are not good candidates for this application, because it is not possible to do displacement washing with these machines. The following vendors tested equipment for this application:

Manufacturer	Equipment
Thermo Black Clawson Inc.	Horizontal belt filter
Baker Hughes	Horizontal belt filter, filter press
Pneumapress Filter Corporation	Pressure filter
Crown Iron Works	Extractor

3.3.4 Test Results

3.3.4.1 Horizontal Belt Filter, Processes P100 and P300

Black Clawson performed bench scale laboratory tests to simulate its Chemi-Washer for both P100 and P300 conditions. Tests were run three times at each condition. Table 7 shows averages of the results for the three test conditions. Black Clawson's complete test report may be found in Appendix C.

Baker Hughes also did laboratory testing to simulate its horizontal belt filter. However, the Baker Hughes report is not presented in sufficient detail to allow any analysis of its data for this equipment.

3.3.4.2 Pressure Filter

Table 8 presents a summary of the Pneumapress hydrolyzate test results.

3.3.5 Equipment Costs

3.3.5.1 Horizontal Belt Filter, P100 Process

Black Clawson provided a budgetary quotation for Chemi-Washers for the P100 application. Three 8-meter-wide x 20-meter-long machines were proposed. Equipment sizing, number of wash stages, and the volume of wash water required for the applicable level of washing efficiency are determined from extrapolation of test results.

HGI estimates the installed cost for this equipment and associated auxiliary equipment to be $27,000,000. A preliminary equipment

list with order-of-magnitude prices for auxiliary items is included in Appendix C.

Baker Hughes provided a budgetary quotation for a horizontal belt washer for the P100 application. Based on test results, Baker Hughes estimates that 1144 gpm (1.1 to 1.2 lb/lb feed slurry) would be required to achieve the target acetic acid residual level. Baker Hughes proposed three Model 4.2 127 EIMCO-Extractor horizontal belt filters with accessories and controls. The estimated installed cost for this equipment and associated auxiliary equipment is $6,300,000.

Table 7
Summary of Black Clawson Hydrolyzate Test Results

Test	No. of Wash Stages	Wash Water (lb water/lb feed)	Feed Slurry Temp (°C)	Wash Water Temp (°C)	Cake Solids (% Dry Weight)	Cake-Washing Effectiveness			
						Glucose Removal Efficiency (%)	Xylose Removal Efficiency (%)	Acetic Acid Removal Efficiency (%)	Residual Acetic Acid (g/kg OD solids)
1	no wash		85		31.58				52.36
2 [1]	1	0.59	85	46	32.00	69.3	69.7	65.5	18.07
3 [1]	1	1.11	85	46	31.58	75.3	76.0	72.4	14.44
4 [2]	1	1.11	85	63	38.76	82.4	82.4	92.0	9.89
5 [1]	4	1.09	85	46	29.84	92.0	92.5	91.7	4.35
6 [2]	4	0.86	85	63	29.75	92.2	92.8	92.5	3.94
7 [2]	4	1.11	85	63	30.42	93.8	94.5	94.5	2.90
8 [2]	4	0.59	85	63	30.73	88.7	89.1	88.4	6.10

[1] Process 100
[2] Process 300

Black Clawson
Summary Test Results for Chemi-Washer

Process P100 level required	Acetic acid 3.3 g/kg in solids*
Process P100 level desired	Acetic acid 1.65 g/kg in solids*
Process P300 level required	95% removal of sugar (X and G)**
Process P300 level desired	98% removal of sugar (X and G)**

* Analysis of test results showed none of the P100 tests produced acetic acid levels at or below 3.3 g/kg. Test 5 produced 4.35 g/kg. More wash stages and/or more wash water are required for greater acetic acid removal.

** Analysis of test results showed none of the P300 tests produced 95% or greater washing efficiency of sugars. Test 7 produced 94.5%and 93.8% washing efficiency for both X and G sugars, respectively. More wash stages and/or more wash water are required for greater sugar removal.

Table 8
Summary of Pneumapress Hydrolyzate Test Results

Test	No. of Washes	Wash Water (lb water/lb feed)	Feed Slurry Temp (°C)	Wash Water Temp (°C)	Cake Solids (% Dry Weight)	Cake-Washing Effectiveness: Glucose Removal Efficiency (%)	Xylose Removal Efficiency (%)	Acetic Acid Removal Efficiency (%)	Residual Acetic Acid[3] (mg/ml)
1	0	0	85	--	54.55	45%	45%	45%	12.6
2[1]	1	0.67	85	47	49.68	57%	57%	54%	10.6
3[2]	1	0.73	85	63	52.58	71%	71%	70%	7.5
4[2]	1	0.91	85	63	51.08	64%	65%	63%	8.7
7	2	1.09	80	no data	55.72	97%	98%	97%	0.5
7A	2	1.09	no data	"	57.09	96%	97%	96%	0.8
8	2	0.87	85	"	54.70	96%	97%	97%	0.8
9	2	0.58	85	"	54.76	95%	96%	95%	0.9

[1] Process 100
[2] Process 300
[3] Value reported from NREL laboratory analysis of samples

Pneumapress
Summary of Test Results

Process P100 level required	Acetic acid 3.3 g/kg in solids*
Process P100 level desired	Acetic acid 1.65 g/kg in solids*
Process P300 level required	95% removal of sugar (X and G)**
Process P300 level desired	98% removal of sugar (X and G)**

* Analysis of test results showed that with two wash stages and 0.58 lb wash water per lb of feed, the residual level of 0.9 g/kg acetic acid was achieved, below both required and desired levels.

** Analysis of Test 8 results showed that 96% glucose and 97% xylose removal was achieved with two wash stages and 0.87 lb of wash water/lb of feed. More wash stages and/or more wash water would be required to obtain 98% sugar removal.

3.3.5.2 Horizontal Belt Filter, P300 Process

Black Clawson provided a budgetary quotation for Chemi-Washers for the P300 application. Four 10-meter-wide x 22-meter-long machines were proposed. Based on the test results and Black Clawson's projections of washing efficiency, to achieve a sugar removal of 95% from the washed solids six wash stages each for each machine are required. The washers would be run with 1105 gallons of wash water per bdst (bone dry standard ton) of feed. Equipment sizing, number of wash stages, and the volume of wash water required for the applicable level of washing efficiency are determined from extrapolation of test results. HGI estimates the installed cost for this equipment and associated auxiliary equipment to be $49,700,000.

Baker Hughes provided a budgetary quotation for a horizontal belt washer for the P300 application. Based on test results, Baker Hughes estimates that 1144 gpm (1.1 to 1.2 lb/lb feed slurry) would be required to achieve 95% sugar removal. Baker Hughes proposed three Model 4.2 127 EIMCO-Extractor horizontal belt filters with accessories and controls. The estimated installed cost for this equipment and associated auxiliary equipment is $6,300,000.

3.3.5.3 Pressure Filter, P100 and P300 Processes

Pneumapress provided a budgetary quotation for these applications. The proposed equipment for the P100 and P300 processes is identical. Three Pneumapress pressure filters would be required with a total of 1080 sq ft of filter area. A simplified block flow diagram, equipment list, and order-of-magnitude capital cost estimate may be found in Appendix D. The estimated installed cost for the Pneumapress pressure filter system is $8,500,000. Cycle times were considered in sizing equipment.

For an application with 50% of the current design capacity (flow rate to the pressure filter in kg/hr), one of the three pressure filters would be eliminated. Each of the remaining two pressure filters would have a throughput capacity of 75% of each of the machines for the 100% capacity case. Hence, the total estimated cost of a liquid solid separation for 50% of current design capacity is $4,151,000.

For an application with 150% of the current design capacity, five pressure filters would be required, four having the same capacity as each of the machines for the 100% case and one having 50% capacity of that capacity. Hence, it is estimated that the total installed cost for liquid solid separation for a plant with 150% of the original design capacity cost would be $10,880,000.

The estimated installed horsepower for a Pneumapress installation for either process P100 or P300 is 1755 hp with an estimated average power demand of 980 kW. Assuming $0.05/kW-hr and 8000 hours of operation per year, the annual electrical energy operating cost is

estimated to be $392,000. Energy costs are expected to vary linearly with the equipment throughput capacity.

3.4 Liquid/Solid Separation First-Stage Hydrolysis, Elevated Pressure/Temperature, Process P100

As an alternative to ambient pressure washing, liquid/solid separation could be provided after the first-stage hydrolysis reactor at an elevated temperature to keep lignin in solution. The expected temperature is 135°C. Washing is expected to be required.

The process objective is to remove as much of the toxins from the solids as practical. The amount of wash water required to accomplish this should be limited to a maximum of 132,000 kg/hr (.58 lb wash water/lb feed) , because this is the total amount of water added to fermentation. If washing proves to be too difficult, cooking could be done at a slightly lower temperature. Potentially, liquid/solid separation could be done without washing.

3.4.1 Test Material

See description of test material in paragraph 3.3.1.

3.4.2 Materials of Construction

Type 316 stainless steel, as recommended for the ambient pressure liquid/solid separation application, is not suitable for 0.4% H_2SO_4 at a temperature of 135°C. While no condition-specific testing has been done, testing was done by InterCorr International for a 0.6% H_2SO_4 at 190°C. Alloy 825 exhibited a corrosion rate of 8 mpy in these tests. Normally, 5 mpy is considered an acceptable corrosion rate. Because H_2SO_4 corrosion is strongly affected by temperature, it is likely that Alloy 825 will function satisfactorily at 135°C. Testing at the actual process conditions of Alloy 825 and testing with other less costly materials are recommended to verify acceptability.

3.4.3 Equipment/Vendor Options

Pneumapress Filter Corporation's pneumatic pressure filter has been identified as equipment that is capable of liquid/solid separation and countercurrent batch washing at pressures and temperatures above the boiling point of the slurry. The Pneumapress filter is capable of high cake solids and good washing with clear filtrate. Countercurrent washing would be done with multiple washes in a batch operation. See paragraph 3.1.3.5 for a schematic of the pressure filter.

3.4.4 Test Results

Testing of a Pneumapress bench scale unit took place at NREL's facilities in Golden, Colorado. No quantitative analyses were done on resultant filtrate or cake solids from these tests. However, from qualitative observation, the results appeared to be promising. NREL has plans to perform some general liquid/solid separation on a pilot scale Pneumapress filter. If those tests are successful and if funds are available, a pilot scale unit would be purchased. This unit would be capable of operating at elevated temperature and pressure conditions.

4. CONCLUSIONS AND RECOMMENDATIONS

4.1 Liquid/Solid Separation After Distillation

The objective of this process function is to dewater post-distillate solids to produce minimum moisture for a fuel burner with maximum heat value. At this writing, Baker Hughes has not yet satisfactorily completed testing that would allow evaluation of its belt filter press and filter press. Table 9 provides a comparison of representative test results with associated estimated installed equipment costs.

Table 9
Post-Distillate Comparison of Test Results and Estimated Direct Capital Equipment Costs

Equipment	Manufacturer	Cake Solids	Estimated Installed Cost
Centrifuge	Alfa Laval	19.9%	$8,800,000
Centrifuge	Baker Hughes	22.9%	No data
Filter press (Option A)	Baker Hughes	34.9%–39.7%	$17,300,000
Filter press (Option B)	Baker Hughes	39.7%–44.4%	$24,500,000
Pressure filter	Pneumapress	88.0% False result	$8,100,000
Belt Filter press	Baker Hughes	no data	No data

should be: low 40s.

Based on the data available from testing done to date, the Pneumapress pressure filter produced dramatically drier cake compared to either Alfa Laval or Baker Hughes centrifuges and at essentially the same installed capital cost of the centrifuges. While full-size centrifuges generally develop somewhat drier cake than laboratory scale units, it is unlikely that they would come close to the cake solids exhibited by the Pneumapress pressure filter testing. The Pneumapress pressure filter is recommended as the best equipment for this application based on testing and equipment evaluation done to date. It is recommended that testing be repeated on a larger scale Pneumapress to confirm the repeatability of these results and optimize the process approach.

4.2 Liquid/Solid Separation First-Stage Hydrolysis, Ambient Pressure/Temperature, Process P100

The objective of this process function is to minimize carryover of acetic acid in hydrolyzate solids. At this writing Baker Hughes has not yet satisfactorily completed testing that would allow evaluation of its horizontal belt filter. Baker Hughes provided a capital cost estimate for its equipment; however, without reliable test results, there is no reliable basis for equipment sizing. Table 10 provides a comparison of representative test results with associated estimated installed equipment costs.

Table 10
P100 Comparison of Test Results and Estimated Direct Capital Equipment Costs

Equipment	Manufacturer	*Wash Water (lb/lb feed)	Acetic Acid Residual (mg/ml)	Estimated Installed Cost
Horizontal belt filter	Baker Hughes	1.1 to 1.2	No data	$ 6,300,000
Horizontal belt filter	Black Clawson	1.1	1.7	$27,000,000
Pressure filter	Pneumapress	0.58	0.9	$ 8,500,000

*Feed as received from hydrolysis

Based on the test results and Black Clawson's projections of washing efficiency, to achieve the threshold acetic acid level of 1.7 g/l in the washed solids, five wash stages each for each machine are required. Per Black Clawson, the washers would be run with 940 gallons of wash water per bdst (bone dry standard ton) of feed. That is the equivalent of 1.1 kg of wash water per kg feed or approximately 300,000 kg/hr. That is far in excess of the limit of 132,000 kg of water per hour. Similarly, Baker Hughes' belt filter uses even more wash water.

The Pneumapress pressure filter produced significantly lower acetic acid residual with 0.58 lb wash water/lb of feed. That is approximately equal to 133,000 kg/hr of wash water. In addition, a Pneumapress installation has substantially lower capital cost than the Black Clawson horizontal belt filter. It is recommended that the Pneumapress pressure filter be selected for use in future work in this application based on superior performance and capital cost for equipment tested to date.

4.3 Liquid/Solid Separation First-Stage Hydrolysis, Ambient Pressure/Temperature, Process P300

The objective of this process function is to remove sugar from hydrolyzate solids. At this writing Baker Hughes has not yet satisfactorily completed testing that would allow evaluation of its horizontal belt filter. Baker Hughes provided a capital cost estimate for its equipment; however, without reliable test results, there is no reliable basis for equipment sizing. Table 11 provides a comparison of representative test results with associated estimated installed equipment costs.

Table 11
P300 Comparison of Test Results and Estimated Installed Equipment Costs

Equipment	Manufacturer	*Wash Water (lb/lb feed)	Sugar Removal Efficiency (% Glucose/% Xylose)	Estimated Installed Cost
Horizontal belt filter	Baker Hughes	1.1 to 1.2	95%	$ 6,300,000
Horizontal belt filter	Black Clawson	1.3	95%/95%	$49,700,000
Pressure filter	Pneumapress	0.87	96%/97%	$ 8,500,000

*Feed as received from hydrolysis

The Pneumapress pressure filter produced significantly higher sugar removal with less wash water and at substantially lower capital cost than the Black Clawson horizontal belt filter. The Baker Hughes horizontal belt filter did not have adequate test data to justify a recommendation by HGI. If Baker Hughes could produce test data to back up its claims, this horizontal belt filter may be worth consideration. However, it does appear that both horizontal belt filters will require high wash water to obtain 98% sugar removal. It is recommended that the Pneumapress pressure filter be selected for use in future work in this application based on superior performance and capital cost for equipment tested to date.

4.4 Liquid/Solid Separation First-Stage Hydrolysis, Elevated Pressure/Temperature, Process P100

All testing for liquid/solid separation at elevated temperature took place at NREL facilities utilizing a bench scale Pneumapress pressure filter and equipment that simulates the Pneumapress pressure filter. NREL plans to evaluate a pilot scale Pneumapress for this process application as well as for other liquid/solid separation process applications including P300, P100 ambient temperature, and post-distillate. If evaluations are successful and funding can obtained, a pilot scale Pneumapress will be considered for purchase so that further liquid/solid separation testing can be done.

APPENDIX A
ALFA LAVAL

HARRIS GROUP INC.
PROJECT NO: 99-10600
REVISION B Mar-07-01

NATIONAL RENEWABLE ENERGY LABORATORY
GOLDEN, COLORADO
CENTRIFUGE - DISTILLATE LIQUID SOLID SEPARATION

MECHANICAL
EQUIPMENT LIST

EQUIP # REV MOTOR #	DESCRIPTION	VENDOR P.O. ISSUED	SIZE CAPACITY HEAD	GEAR RATIO	EQUIP STATUS	HORSEPOWER RPM VOLTS	ENCLOSURE FRAME MODEL NO.	REMARKS
C-101	CENTRIFUGE No. 1	ALFA LAVAL	Mod P7600			300 HP		316 SS WETTED COMPONENTS
C-102	CENTRIFUGE No. 2	ALFA LAVAL	Mod P7600			300 HP		316 SS WETTED COMPONENTS
C-103	CENTRIFUGE No. 3	ALFA LAVAL	Mod P7600			300 HP		318 SS WETTED COMPONENTS
C-104	CENTRIFUGE No. 4	ALFA LAVAL	Mod P7600			300 HP		319 SS WETTED COMPONENTS
C-105	CENTRIFUGE No. 5	ALFA LAVAL	Mod P7600			300 HP		316 SS WETTED COMPONENTS
C-106	CENTRIFUGE No. 6	ALFA LAVAL	Mod P7600			300 HP		318 SS WETTED COMPONENTS
C-107	CENTRIFUGE No. 7	ALFA LAVAL	Mod P7600			300 HP		319 SS WETTED COMPONENTS
CV-101	DISCHARGE CONVEYOR No. 1					10 HP		
CV-102	DISCHARGE CONVEYOR No. 2					10 HP		
P-101	CENTRIFUGE FEED PUMP No. 1	GOULDS	MOD 3196 3X4-10 200 GPM 70 FT TDH			10 HP 1780 RPM		IRON CASING W/316 SS TRIM
P-102	CENTRIFUGE FEED PUMP No. 2	GOULDS	MOD 3196 3X4-10 200 GPM 70 FT TDH			10 HP 1780 RPM		IRON CASING W/316 SS TRIM
P-103	CENTRIFUGE FEED PUMP No. 3	GOULDS	MOD 3196 3X4-10 200 GPM 70 FT TDH			10 HP 1780 RPM		IRON CASING W/316 SS TRIM
P-104	CENTRIFUGE FEED PUMP No. 4	GOULDS	MOD 3196 3X4-10 200 GPM 70 FT TDH			10 HP 1780 RPM		IRON CASING W/316 SS TRIM
P-105	CENTRIFUGE FEED PUMP No. 5	GOULDS	MOD 3196 3X4-10 200 GPM 70 FT TDH			10 HP 1780 RPM		IRON CASING W/316 SS TRIM
P-106	CENTRIFUGE FEED PUMP No. 6	GOULDS	MOD 3196 3X4-10 200 GPM 70 FT TDH			10 HP 1780 RPM		IRON CASING W/316 SS TRIM
P-107	CENTRIFUGE FEED PUMP No. 7	GOULDS	MOD 3196 3X4-10 200 GPM 70 FT TDH			10 HP 1780 RPM		IRON CASING W/316 SS TRIM
T-101	FILTRATE TANK	GOULDS	12' -6" DIA X 13' FT H 12,000 GAL					316 SS CONSTRUCTION
P-103	FILTRATE PUMP	GOULDS	MOD 3196 6X8-13 1300 GPM 70 FT TDH					316 SS CONSTRUCTION

Alfa Laval

February 11, 2000

John Lukas
Harris Group, Inc.
PO Box 3855
Seattle, WA 98124-3855

Dear John:

I am enclosing the centrifuge demonstration laboratory test report for National Renewable Energy Lab.

Let me know if you have any questions. I look forward to talking to you soon.

Sincerely yours,

Richard J. Weeks

Richard J. Weeks
Regional Sales Manager

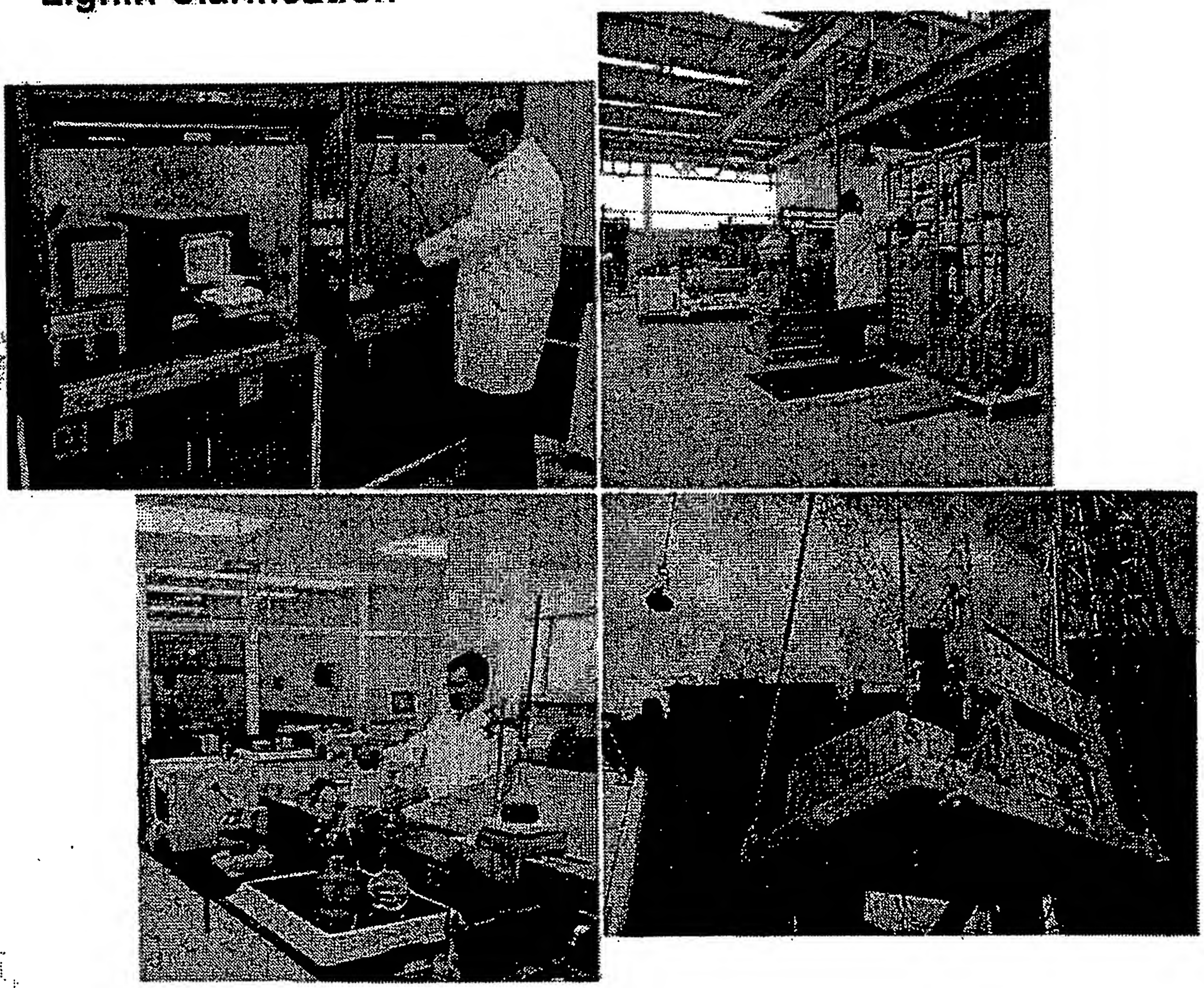
Enclosure

Alfa Laval Separation Inc.
1202 Grant Avenue, Suite E, Novato, CA 94945
Phone: (415) 897-8942 Fax: (415) 897-3263
www.alfalaval.com

Centrifuge Demonstration Laboratory Test Report for:

Harris Group, Inc
National Renewable Energy Lab

Lignin Clarification



Alfa Laval

Alfa Laval

CENTRIFUGE DEMONSTRATION LABORATORY TEST REPORT

Harris Group, Inc. / National Renewable Energy Lab
2901 Third Avenue
Suite 600
Seattle, WA 98121

TITLE: ***LIGNIN CLARIFICATION***

TEST OBJECTIVES:

Harris Group and the National Renewable Energy Lab (NREL) are interested in performing a separation on the bottoms from an evaporator. The feed slurry contains lignin and cellulose suspended in water. NREL wants to obtain a cake with minimal moisture content to use as fuel in a burner. The clarified liquid is to be recycled back into a fermentation process. The exact specifications for cake moisture and effluent clarity are unknown at this point. The process is to be performed at 180°F and full scale throughput requirements are approximately 1100 gpm.

CONCLUSIONS:

A 1.5 minute spin at 1,500 x G dropped the concentration of insolubles in the feed slurry from 23% by volume to 0.07% by volume, yielding a recovery of 99.7%. This indicates that the application fits well within the capabilities of a decanter type centrifuge. In addition, cake samples collected from spin testing contained 19.9% total solids. This value can be expected to increase in a continuous decanter.

As a result, further testing with a decanter is recommended. This would serve both to provide scale-up data and to confirm the feasibility of the continuous process.

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Alfa Laval

PROCEDURE:

On January 4th, 2000, a five-gallon pail of lignin slurry was received at Alfa Laval's Centrifuge Demonstration Laboratory (CDL) in Warminster, PA.

On January 20th, 2000, a representative mixed slurry sample was collected from the five-gallon pail and transferred to a 1000 ml beaker. The beaker was heated to 180°F while being stirred continuously. A series of tests were then performed using a Hotspin heated bench top test tube centrifuge operating at 1,500 x G and 86°C. Each test involved a pair of 10 ml samples being spun for a defined time interval. The resulting supernatant liquid was analyzed for residual insolubles, and the volume and consistency of the sedimented solids was noted. Tests were performed at time intervals of 2.0, 1.5, 1.0, and 0.5 minutes. A 3.0 minute spin in a Gyrotester bench top centrifuge operating at 10,000 x G was used to analyze supernatant samples for remaining insolubles.

The following tests were performed in addition to those described above: feed total solids, supernatant total solids from a 1.5 minute spin, feed viscosity, cake total solids, and liquid total dissolved solids.

DISCUSSION:

Results from this Separation Study confirm that this application is well suited to a decanter type centrifuge. A 1.5 minute spin at 1,500 x G yielded a 99.7% recovery based on volume content of insolubles. The cake from the spin tests contained 19.9% total solids, which in general is a conservative approximation of the cake obtained from a continuous decanter. The solids separate and compact well, indicating that there should not be an issue with either resuspension inside the decanter or scrolling the cake out of the bowl.

The feed slurry contained approximately 25% by volume total suspended solids. The solids were present in two distinct layers, one made up of coarse grit and one made up of soft fines. The feed slurry was analyzed to have 7.26% by weight total solids, and the effluent from a 1.5 minute spin contained 4.26% total solids. A total dissolved solids analysis was also performed, yielding 3.54% by weight. The feed slurry viscosity at 180°F was measured to be 13.5 cP.

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Based on the outcome of this trial, further testing with a decanter centrifuge is recommended. Such a test would confirm that the process is feasible on a continuous basis while providing data to scale up to a full size decanter.

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Alfa Laval

HOTSPIN EVALUATION

(RCF X G = 1,500)

REPORT #: CDL 2529

DATE: Jaurnary 20, 2000

CLIENT: Harris Group / NREL

APPLICATION: Lignin clarification

Spin Time (min)	0.5	1.0	1.5	2.0		3.0 Gyrotester
Slurry as Received	Opaque, brown slurry. No visible insolubles or floaters.					
Clarified Liquid Appearance	Very cloudy	Cloudy	Slightly cloudy	Clear		Clear
%V/V Insol. in Clarified Liq. *	0.6	0.12	0.07	0.02		
Sedimented Solids (%v/v) Description(Firm, Soft)	28-29 Soft fines Firm grit	21-22 Firm	22-23 Firm	22-23 Firm		~25 Firm, 2 solids layers - fines and grit

* Spun In Gyrotester 3 Min.

Comments:

All testing performed at 180°F

Feed = 7.21% T.S., 13.5 cP

Supernatant from 1.5 minute spin = 4.26% T.S.

Clarified liquid = 3.54% T.D.S.

Cake sample = 19.9% T.S.

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APPENDIX B
BAKER HUGHES

H

BAKER HUGHES

Baker Process

669 West 200 South
P.O. Box 300
Salt Lake City, UT 84110-0300
Tel 801.526.2329
Fax 801.526.2435

February 5, 2001

Lynn Montague
Harris Group
100 Denny Way
Suite 800
Seattle, Washington

Dear Lynn:

Liquid Solid Separation after Distillation:

Filter press results produced the highest cake solids for the hydrolysis material. The results are summarized below.

Table 1
Filter Press Results
Hydrolysis

Test #	Filtration Time minutes	Membrane Time minutes	Filtration Pressure psig	Membrane Pressure psig	Cake Solids, wt%	Dry Cake Density, lbs/ft^3
P-1	30	15	100	225	32.95	10.1
P-2	45	N/A	100	N/A	34.87	11.4
P-3	62	28	100	225	44.4	33.2
P-4	62	8	100	225	39.7	28.2
P-6	120	N/A	100	N/A	37.3	26.5

Filter Sizing for the hydrolysis material is:

Table 2
Filter Press Sizing for Hydrolysis

	Rapid discharge 2 x 2 meter plates	ALP Discharge 2 x 2 meter plates	Rapid discharge Membrane 2 x 2 meter plates	ALP Discharge Membrane 2 x 2 meter plates
Feed Rate, lbs. slurry/hr	592,533	592,533	592,533	592,533
Solids Concentration, wt%	11.7%	11.7%	11.7%	11.7%
Solids Rate, lbs./hr	69,326	69,326	69,326	69,326
Dilution Concentration, wt%	11.7%	11.7%	11.7%	11.7%
Cake Density, lbs/ft^3	26.5	26.5	26.5	26.5
Filter Volume required, ft^3/hr	2616	2616	2616	2616
Filtration Time, minutes	62	62	62	62
Membrane Time, minutes	0	0	15	15
Air Blow Time, minutes	0	0	0	0
Discharge Time, minutes	3	19	3	18
Cloth/Flood Wash, minutes	2	2	2	2
Cycle Time, minutes	67	83	82	97
Cycles/hour	0.90	0.72	0.73	0.62
Air Req'd, scfm/ft2	0.00	0.00	0.00	0.00
Feed Rate, gpm/filter (average filtration time))	176	273	181	256
Filter Volume Required, ft^3/cycle	2921	3619	3575	4229
Volume, ft^3/chamber	5.65	5.65	5.65	5.65
Number of Chambers	517	641	633	749
Number of Chambers/Filter	103	160	106	150
Number of Filters	5.0	4.0	6.0	5.0

Sizing is provided based on using the EIMCO automated filter discharge, where the plates are discharged within 3-4 minutes, or using a conventional discharge (ALP –Automatic Large Press) where the discharge is usually 6 seconds/plate. The difference is in the total number of chambers. The filters will require four (4) units in each case. These are 225 psig filters. The use of 225 psig will be necessary to offset the difference in final cake solids obtained with 100 psig filtration and 225 psig membrane operation. Sizing is provided for both recessed and membrane filters. The filter size is limited 120 chambers or less for the EIMCO discharge mechanism and lee than 180 chambers for the standard discharge. The number of filters is adjusted to meet these criteria

1. Centrifugation was tested, but the following additional information should be provided:

- Presentation and evaluation of test data and results

Table 3
Centrifuge Data

Harris Group, Low Solids Sample Centrifuge Tests

Test #	RPM	Time (min)	Tare	Feed Weight	Cake Thickness mm	Rod Depth	% Penetr	Calc. Cake % Solids
1A	1500	3	38.79	48.84	8	2.5	31.3	24.4
1B	1500	3	38.73	48.88	10.0	4.5	45.0	24.0
2A	2000	2	38.79	48.91	10	7	70.0	25.1
2B	2000	2	38.73	48.81	10	5	50.0	24.9
3A	2000	4	38.79	48.96	10	2	20.0	25.6
3B	2000	4	38.73	48.84	10	3	30.0	25.6
4A	1000	4	38.79	48.91	11.5	8.5	73.9	23.4
4B	1000	4	38.73	48.81	11.5	7	60.9	23.5
5A	1000	2	38.79	48.89	13.5	11.5	85.2	22.1
5B	1000	2	38.73	48.83	13.5	10.5	77.8	22.3
6A	3000	2	38.79	48.87	10	2	20.0	27.0
6B	3000	2	38.73	48.85	10	2	20.0	26.6
7A	2900	1	38.79	48.93	10.5	3.5	33.3	26.6
7B	2900	1	38.73	48.88	11.5	3	26.1	25.9
8A	2000	1	38.79	48.86	11	4	36.4	24.4
8B	2000	1	38.73	48.90	12	6	50.0	24.2
9A	1500	2	38.79	48.69	12	8	66.7	23.0
9B	1500	2	38.73	48.88	11.5	7.5	65.2	23.2

The main point in these data is that the final solids content is 25-wt% or less. This will probably be to low for incineration.

The centrate solids will be in the range of 2500-3500 ppm, as shown below. Some of the tests produce lower solids , however because of the high dissolved solids in the filtrate, compared to the suspended solids these numbers actually be greater in actual operation. These solids would cause problems in water treatment or recovery operations.

Baker Process experience in the use of centrifuges in this type of process (wheat, barley, sorghum) was that land application of the final centrate was necessary to make a cost effective treatment.

Table 4
Distillate Centrate Solids

Centrate Solids DS = 2.84 wt %

Sample #	Tare Wt.	Dry Wt.	Wet Wt.	TS	TSS
1A	1.02	1.23	8.22	2.92	0.08%
1B	1.01	1.23	8.34	3.00	0.17%
2A	1.01	1.23	8.43	2.96	0.13%
2B	1.01	1.22	8.39	2.85	0.01%
3A	1.01	1.23	8.43	2.96	0.13%
3B	1.00	1.22	8.38	2.98	0.15%
4A	1.01	1.22	8.17	2.93	0.10%
4B	1.01	1.22	8.25	2.90	0.06%
5A	1.01	1.23	7.96	3.17	0.33%
5B	1.01	1.23	8.04	3.13	0.30%
6A	1.01	1.23	8.5	2.94	0.10%
6B	1.00	1.23	8.56	3.04	0.21%
7A	1.00	1.23	8.55	3.05	0.21%
7B	1.00	1.23	8.54	3.05	0.22%
8A	1.01	1.23	8.37	2.99	0.15%
8B	1.01	1.23	8.38	2.99	0.15%
9A	1.01	1.23	8.11	3.10	0.27%
9B	1.01	1.23	8.32	3.01	0.17%

- Recommendations

At the present the centrifuge is not recommended because it does not produce sufficient solids for incineration and the final centrate will need treatment and/or disposal.

- Equipment sizing if this equipment is recommended for this application

Not required

- Budgetary estimate if this equipment is recommended for this application

Not required

2. Belt press was tested, but the following additional information should be provided:

- Presentation and evaluation of test data and results are listed below.

Table 5

Zelco 7565

EIMCO PROCESS EQUIPMENT COMPANY

EIMCO- BELTPRESS BENCH TEST DATA AND SIZING

COMPANY: Harris Group DATE: 2/2/2000

MATERIAL: TVA Wood Bark (poplar) Distillate BY: MRP

TEST NO.		1	2	3	4	5	6	7
	pH	5.40	8.50	8.50	8.50	8.50	8.50	8.40
	Density g/cc	1.01	1.01	1.01	1.01	1.01	1.01	1.01
	% Passing 325 mesh							
	Solids Conc. wt%	3.98	3.98	3.98	3.98	3.98	3.98	3.98
	Identification	Perc-765	Perc-765	Perc-765	Perc-765	Perc-765	Perc-765	Perc-765
	Concentration wt%	0.01	0.02	0.02	0.02	0.02	0.02	0.01
	Addition mls	50	40	40	40	40	45	90
	Dosage lbs/ton	1.48	1.32	1.32	1.32	1.32	1.49	1.49
	Mixing Rotations/sec	2	2	2	2	2	3	2
Filtration Media		FE4572	FE4572	FE4572	DB-2130	FE4572	FE4572	FE4572
Sample Size mls		167	300	300	300	300	300	300
	Drainage Time sec	120	60	100	90	60	60	65
	Filtrate Vol. ml	310	260	255	265	270	280	325
	Filtrate Distrib.	poor	poor	poor	poor	poor	poor	fair
	G.D. Soil Conc. wt%	16.33	16.30	16.30	18.27	19.23	19.00	17.52
	Filtrate Conc. wt%	0.65	0.27	0.17	0.58	0.34	0.25	0.29
Calculated Belt Speed ft/min		14.5	14.5	8.7	9.7	14.5	14.5	13.4
Calculated Feed Rate gpm/meter		10.7	19.3	11.6	12.9	19.3	19.3	17.8
Calculated Solids Rate lbs/hr/m		216.9	389.6	233.7	259.7	389.6	389.6	359.6
	Pressure psig	BDP	BDP	BDP	BDP	BDP	BDP	BDP
	P-Roll #			40			40	
	Cake Thickness mm	8.7	9.8	9.3		8.8	6.6	8.3
	Cake Solids wt%	26.36	29.22	34.54		25.96	29.82	27.04
Discharge Solids Rate lbs/hr/m		1500.0	1910.7	1320.2		1500.6	1317.0	1368.0
Calculated Feed Rate gpm/meter								

The belt press results show that the solids can be dewatered to 26-29 wt% with a belt press filter. A polymer dosage of 1-2 lbs/ton will be required. In general the filtrate clarity is poor, with solids of 0.25 wt% or greater. In commercial operation the belt wash water will increase the solids by removing residual solids from the belts. Typically the combined belt wash water and the filtrate will contain 0.5-1.0 wt% for this application.

The tests were run using a dilute feed concentration, 3-4 wt% compared to 11.7 wt% for design. The ability to flocculate the solids may be impacted by this difference and either dilution of the feed, greater mixing intensity, and/or increased polymer dosages may result.

- Recommendations that reference test results and data

Belt Press filters may be used. Based on solids rates the equipment would require 90 meters of belt width, (30 3 meter machines). The filtrate will require treatment or disposal with 0.25-1.0 wt% solids in this fluid. Final cake solids will be 25-29 wt%.

- Equipment sizing if this equipment is recommended for this application

Based on the final solids content and the filtrate solids belt presses are not recommended for this application.

- Budgetary estimate if this equipment is recommended for this application.

Not required

Liquid Solid Separation after 1st Stage Hydrolysis

1. Belt filter was tested, but the following additional information should be provided:

- The conclusions and recommendations in the October report needs some clarification and some additional information -

- How much wash water is required is required to achieve the target sugar removal level of 95%.

The quantity of wash water required is 1144 gpm using two stages of counter-current wash. This is 4.43 lbs/lb. solid or about 1.1-1.2 lbs/lb. feed slurry.

- There appears to be some problem with the unit in the conclusion regarding acetic acid removal. For example the report states that the initial acetic acid content was 23,000 g/kg in an unwashed cake. That equates to 23 kg/kg, which isn't possible. Please check result to see if it effects your analysis of the result and equipment sizing.

The acid content listed in the design criteria is for 1.4-2.2 wt% acid. This would represent concentration of 14-22 g/kg. The analysis was 23 g/kg. During the test evaluation the concentration was misinterpreted as 23,000 g/kg and this should have been as mg..

The targets for acetic acid are:

Targets	Cake Moisture, wt%	Liquor Concentration, g/kg
AA maximum limit - 3.3 g/kg	67	4.92
AA desired limit - 1.65 g/kg	67	2.46
AA future limit - 7.1 g/kg	67	10.6

The targets are assumed to be on a wet cake basis and the concentration in the liquor is calculated to allow evaluation with the wash correlation.

Table 6
Process 100/300 vacuum filter sizing

Error! Not a valid link.

These data are based on application of a two wash stages using two displacements of water per stage. This is slightly more than the maximum required for make-up (989-1074 gpm) If additional wash water is used then the recovery of sugar and acid will increase slightly. This design is sufficient to meet the targets for acetic acid in the final cake.

The sugar recovery will be 95% or greater. To increase the sugar recovery to 98% would require 4 displacements (2204 gpm) applied as 3 stages of wash. This would result in the filtration rate decreasing by nearly 50%. To use the minimum water and achieve 98% recovery would require 4-6 displacements to approach the desired value. At six displacements the filtration rate would be 16.4 lbs/hr/ft2 compared to 32.3 at the two displacement two stage scenario.

- Provide a budgetary estimated for the recommended equipment.

Gene Haas will provide this under separate cover.

2. Filter press was tested. No test results were presented to support recommendations or conclusions.

Sincerely,

Vaughn Weston
Process Engineer
801-526-2329

Table 6
Process 100/300 vacuum filter sizing

	EIMCO-Extractor HBF 100 process	EIMCO-Extractor HBF 300 process
Feed Rate, lbs dry solids/hr	129154	129180
Design Filter Area, ft^2	4000.84	4001.76
Filter Cloth	POPR950	POPR950
Vacuum Level, in.Hg.	20.00	20.00
Air Rate, cfm/ft2 @ Vacuum @ Sea Level	0.78	0.99
Feed Concentration, wt%	30.00	30.00
Feed Temperature, °C	20	20
Wash Temperature, °C	60	60
Slurry pH	7-9	7-9
Design Cake Thickness, mm	15.00	15.00
Design Cake Weight, lbs/ft2	0.89	0.89
Design Cake Moisture, wt%	66.9%	66.9%
Fraction Remaining, R, final conc/initial conc.	0.08	0.08
Wash Displacements, N volume wash/volume cake liquor	2.00	2.00
Number of Wash Stages	2.00	2.00
Wash Rate, gpm	1144	1144
Filter Submergence, %		
Design Influent Solute Concentration, mg/l:	23000.00	23000.00
Design Solute Concentration ppm dry solids basis	5000	5000
Design Solute Concentration, ppm wet cake basis	1650	1650
Solute Concentration Final Cake Liquor	2257	2257
Filtration Rate, lbs/hr/ft^2 (incl 0.8 Scale up)	32.28	32.28
1 Form Time, seconds	24	24
2 Wash Time, seconds	38	38
3 Dry Time, seconds	16	16
Cycle Time, Minutes	1.64	1.64
Correlation Constants		
k_1 Cake Thickness (W=k$_1$T)	0.08	0.08
k_2 Form Time (W=k$_2$T$^{0.5}$)	0.15	0.16
k_3 Wash (W/Ww=k$_3$*Wash Time)	0.02	0.02
k_4 Dry (Dry Time/W)	18.44	18.44
n Slope (W=k$_2$T^n)	0.50	0.50

These data are based on application of a two wash stages using two displacements of water per stage. This is slightly more than the maximum required for make-up (989-1074 gpm) If additional wash water is used then the recovery of sugar and acid will increase slightly. This design is sufficient to meet the targets for acetic acid in the final cake.

BAKER HUGHES

BAKER PROCESS

REPORT OF INVESTIGATION

Dewatering and Washing of Hydrolyzed Biomass

for

Harris Group Inc.

Seattle, WA

JOB #:

BY

Mark Peterson/Vaughn Weston

Process Engineer

CHEMICAL AND INDUSTRIAL
APPLICATIONS & PROCESS TECHNOLOGY
SALT LAKE CITY, UTAH
August 2000

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SUMMARY

Dewatering tests were conducted on two samples of material from a Biomass conversion process. The first stage acid hydrolyzed material was filtered and washed using horizontal belt vacuum filters and pressures filters. The results indicate the material can be filtered from about 26 wt% solid to produce a final cake containing about 33 wt% solids. The material will need to be diluted to about 20-21 wt% to provide a feed that can be delivered to the filters. The wash and/or filtrate liquors may used for this dilution.

Filter sizing is provided in this report.

The second sample, the distillate was dewatered using pressure filters, centrifuges, and twin belt (belt press) filters. These results have not be evaluated at this time and will be provided in a final version of this report.

INTRODUCTION

Harris Group Inc. submitted three samples for the following process operations:

- Centrifugation
- Vacuum Filtration
- Pressure Filtration
- Belt Press (Twin Belt) Filtration (Expression)

The samples represent two treatment conditions. The first sample was hydrolazate containing acid and sugar residue that requires dewatering and washing to recover the acid and sugar. The third sample was a complement to this sample to provide liquid to re-slurry the primary sample.

The second sample was the hydrolazate residue which requires dewatering for final disposal by incineration. Maximum cake solids are desired for this material. Cake washing was not required for this material.

Treatment Objective

The target treatment objectives are listed in Table 1.

Table 1
Treatment Objectives

PROCESS TARGET OBJECTIVES				
Product Stream:		Liquids and Solids		
		units		
Solids Targets			Process 100	Process 300
	Solids Concentration	wt%	60	63.9%
	Solute Concentration (dry basis)	wt%		
Liquid Targets				
	Suspended Solids	wt%	0.1	0.9

DESIGN CRITERIA

Table 2 lists the basic design criteria provided by Harris Group Inc.

Table 2
Process Design Criteria

PROCESS DESIGN:	units	Process 100	Process 300
FEED RATE	gpm	869	869
SOLIDS CONCENTRATION:	wt%	26.1%	26.1%
SOLIDS RATE	lbs/hr	129,180	129,180
Total Suspended Solids, (TSS)	wt%	26.1%	26.1%
Total Dissolved Solids, (TDS)	wt%	9.1%	9.1%
Solids Density	g/cc		
Slurry Density	g/cc	1.138	1.138
Liquor Density	g/cc	0.98	0.98
pH:	units		
Temperature:	°F	214	214
Chemical Analysis:(Specify)			
Acid (sulfuric)	wt%	1.5-2.4	1.5-2.4

TEST PROCEDURES

Laboratory tests were conducted using standard procedures developed by Baker Process (formerly EIMCO PEC) during the past 50 years. Specific written procedures will be provided upon request of the client.

Solids analysis were performed by drying at 102 oC until a stable weight was obtained. This was due to the high dissolved salt content in some of the samples.

DISCUSSION OF RESULTS

SAMPLE CHARACTERIZATION

The samples was analyzed for the following parameters as listed in **Error! Not a valid link.** ;

Table 3
Sample Characterization

Parameter	Units	Sample 1	Sample 2	Sample 3
pH:	units	1.6	5.0	1.6
Temperature:	°C	20	20	20
Total Suspended Solids (TSS):	wt%	26.4%	3.84%	7.89%
Total Solids (TS):	wt%	36.0%	6.51%	18.81%
Total Dissolved Solids (TDS):	wt%	N/A	3.84%	10.95%
Slurry Density:	g/ml	1.010	1.010	1.080
Solids Density:	g/cc			
Liquor Density:	g/ml	1.050	1.000	1.050

VACUUM FILTRATION TEST RESULTS

Vacuum filtration tests were conducted to determine the filtration requirements to dewater a slurry containing Distillation Solids. The objectives of the filtration tests included;

- Determine the cake filtration rate
- Determine the cake washing rate
- Determine the cake drying rate
- Determine the air flow and related vacuum requirements
- Determine the filter requirements to process the material described in the design criteria.

VACUUM FILTRATION DISCUSSION OF RESULTS

The test procedures and data correlation's discussed in the following section are based on methods Baker Process (EIMCO) has developed to optimize the evaluation and design of filtration equipment. This report is prepared using these correlation's. The data correlation's are used to determine design relationships and these are then used to calculate the function times and filtration performance.

VACUUM FILTRATION TEST PROCEDURES

Vacuum filtration tests were conducted using laboratory procedures developed by BAKER Process to determine the various filter functions. A copy of the test procedures is available for reference. This may be requested from Baker Process if not submitted with the report. The data collected are correlated using various relationships to determine the design parameters for the vacuum filter. These data are described in the following discussions.

Cake Weight vs Cake Thickness

Figure 1 shows the relationship between the filter cake weight, mass dry solids/filter area. The data can be represented by Equation 1.

Equation 1

$$W = k_1 T$$

Where:

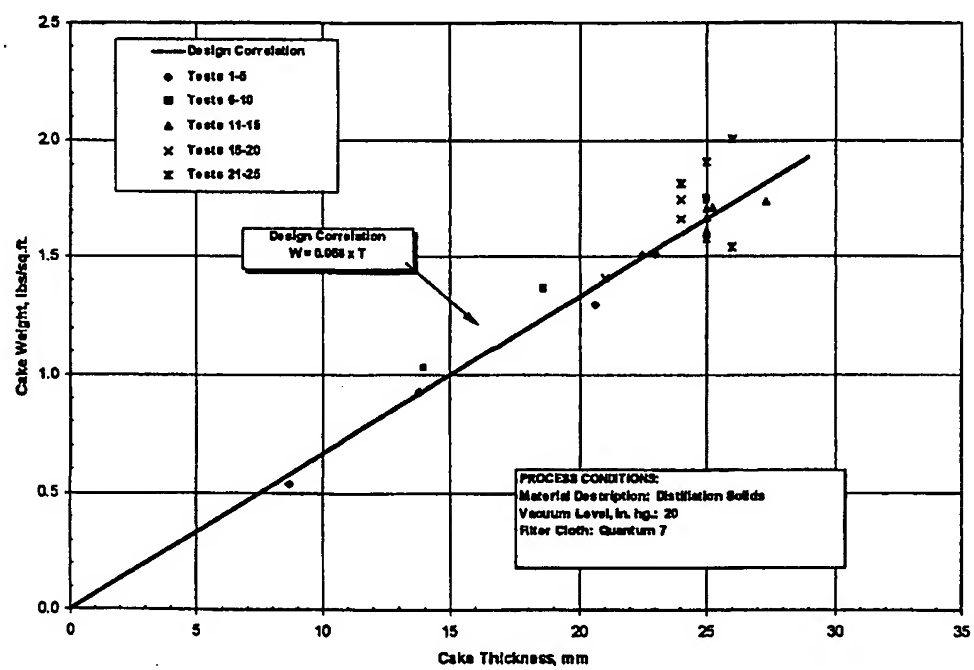
W = Cake Weight, mass / unit area (lbs / ft^2, kg / m^2)

k_1 = Correlation Constant for design

= Cake Thickness, mm

The correlation constant are listed in the Figure for the design correlation line(s) shown. The line labeled as Design Correlation will be used for filter design.

Figure 1
Cake Weight vs Cake Thickness



The slope constant from the Design Correlation line is entered into the Filter Sizing summary table where the cake weight for the selected design cake thickness is calculated and then used to determine the filter function times from the rate correlation's.

The cake thickness is selected to provide acceptable discharge from the operating filter. The minimum cake thickness will vary depending on the type of filter and the discharge characteristics of the material. The minimum cake thickness that can be discharged from the filter will produce the maximum cake filtration rate. The minimum thickness' for the equipment being considered are listed in Table 4:

Table 4
Discharge Cake Thickness

Filter Type	Discharge Mechanism	Minimum Cake Thickness for Discharge
EIMCO EXTRACTOR® horizontal belt Filter	Belt Discharge	3 mm (1/8")

For this application a 1 inch (25 mm) cake is selected because the cake formation time is relatively short and there is a need to evenly distribute the slurry across large filters will be necessary. Thinner cakes will make this process more difficult.

CAKE FORMATION RATE

The cake formation rate is determined by plotting the dry cake weight with respect to the cake form time, using a log-log relationship. The data correlation can be described by the general equation shown in Equation 2 which is derived from filtration theory.

Equation 2

$$W = k_2 \Theta_f^{0.5}$$

Where:

W = Cake Weight, mass dry salt free solids / unit filter area

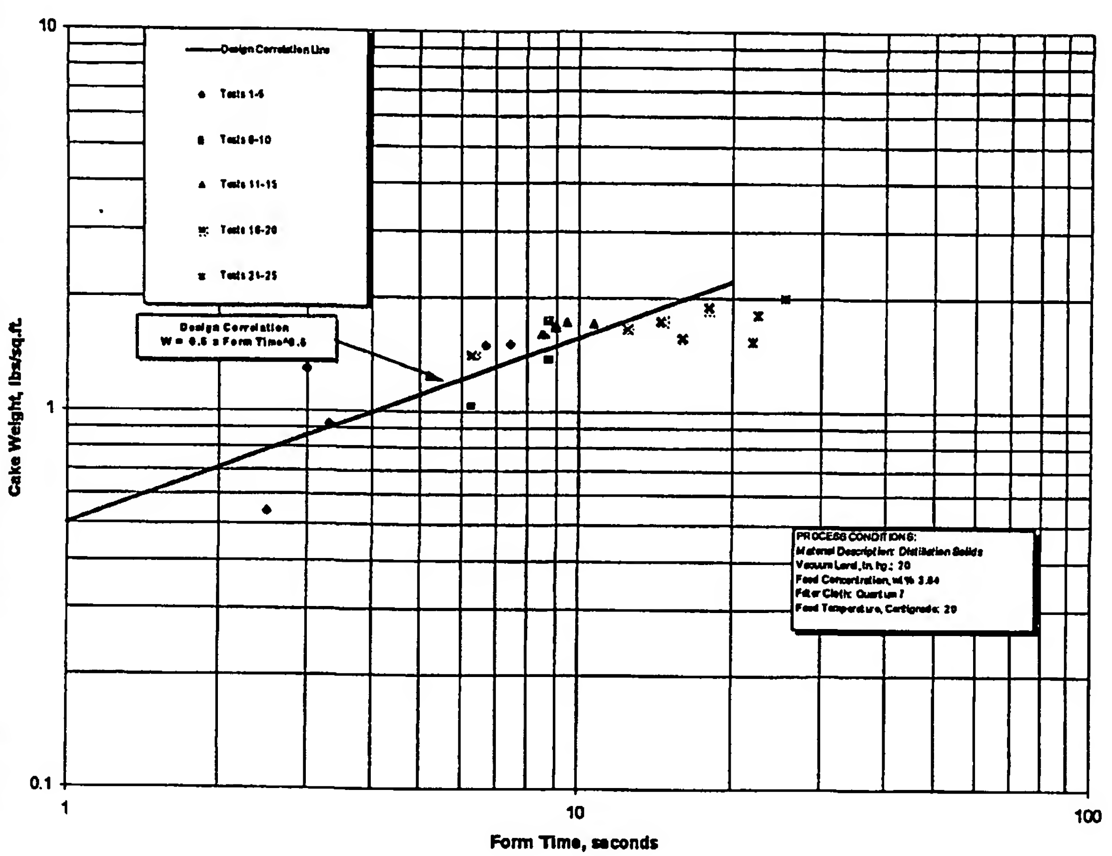
k_2 = Intercept constant at Form Time of 1

Θ_f = Form Time, time units of seconds or minutes

0.5 = Slope predicted from filtration theory

The test data are shown in Figure 2. The primary data that is used for design is represented by the design correlation line. The equation for this line is listed in the figure. The constants from this equation are entered in Filter sizing Summary Table and used to calculate the cake form time for a selected cake thickness.

Figure 2
Cake Weight vs Form Time



CAKE WASHING RATE

The cake washing rate is determined by plotting a wash correlation factor with respect to the wash time. The wash correlation factor is the product of the cake weight, W (mass/unit area), and the wash volume V_w(volume/unit area) i.e. W x V_w (lbs/ft^2-gal/ft^2;kg/m^2-l/m^2) The cake weight is used as a method of normalizing the data for variations in the cake resistance. If the cake weight changes, then the resistance also changes resulting in an expected proportionate change in the wash time. Similarly, If the wash volume is changed the wash time is also expected to change proportionately. The data are represented by Equation 3. The test data are shown in Figure 3.

Equation 3

$$WV_w = k_3 \Theta_w$$

Where:

W = **Cake Weight, mass salt fess solids / unit area**

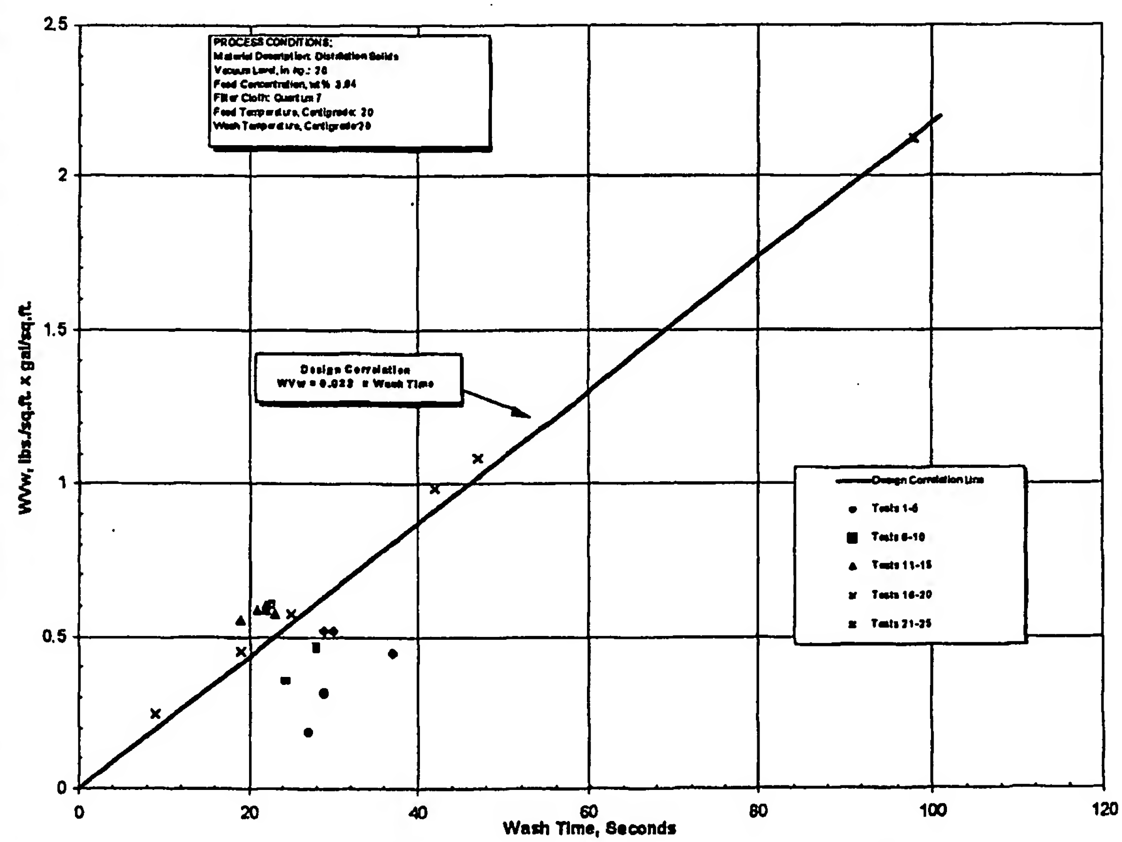
V_w = **Wash Volume, volume / unit area**

k_3 = **Slope constant for correlation**

Θ_w = **Wash Time, units of seconds or minutes**

This is one of several correlation's that may be used for this purpose The wash time is plotted on the x-axis to provide consistency. The function times in all of the correlation's are plotted on this axis.

Figure 3
WVw vs Wash Time



The data will generally follow a linear relationship. There are several reasons why the correlation may not be linear including;

- Changes in the wash water temperature.
- Removal of cake liquor which has different viscosity than the wash liquor.
- Cake compacts as the mother liquor is removed from the cake causing the cake to compress and become more resistant.
- Cake cracking occurs allowing the wash liquor to short circuit the cake.

The design correlation line shown in the figure is placed to best represent the data. Where the data does not provide a straight line relationship the design line is placed such that the expected design condition will be on or near the design line. This procedure allows the design values to automatically be entered into the filter summary table.

CAKE DRYING RATE

The cake drying rate is determined by plotting the final cake moisture content, or the cake liquor content as a function of, or with respect to, a correlation factor or approach factor. An empirical factor that includes the pressure drop across the cake (vacuum level), Air flow, cake weight, drying time, and liquor viscosity was developed. The relationship is shown in Equation 4.

Equation 4

$$\text{Cake Moisture, wt\%} \int \frac{\Delta P}{W} x \varpi \ x \frac{\Theta_d}{\mu}$$

Where:

ΔP = Pressure Differential (Vaccum Level)

W = Cake Weight, mass / unit area

ϖ = Air Flow, Volume / unit time / unit area

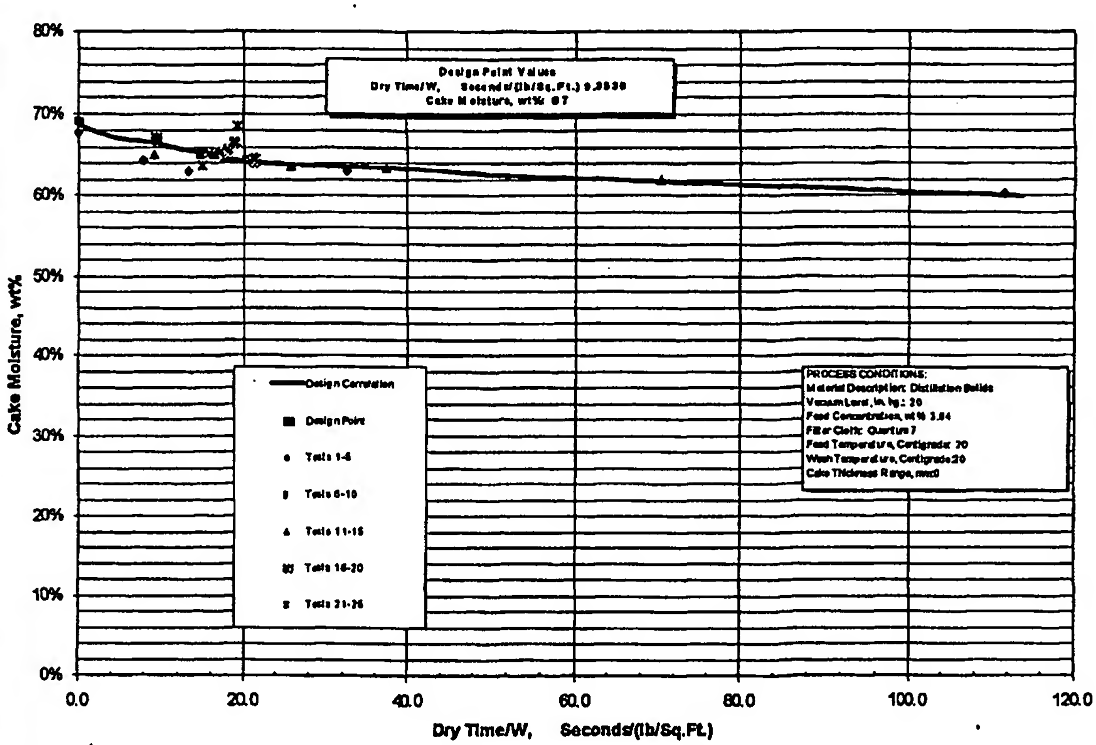
Θ_d = Dry Time, unit time

μ = Liquor Viscosity,

In many applications several of the variables will remain constant and the relationship can be simplified to that in Equation 5. The dry time at any specific moisture content can be calculated from the value of the correlating factor by factoring with the design cake weight. The test data are shown in Figure 4.

Figure 4

Cake Moisture vs θ_d/W



Equation 5

$$\text{Cake Moisture, wt\%} \int \frac{\Theta_d}{W}$$

Where:

W = Cake Weight, mass / unit area

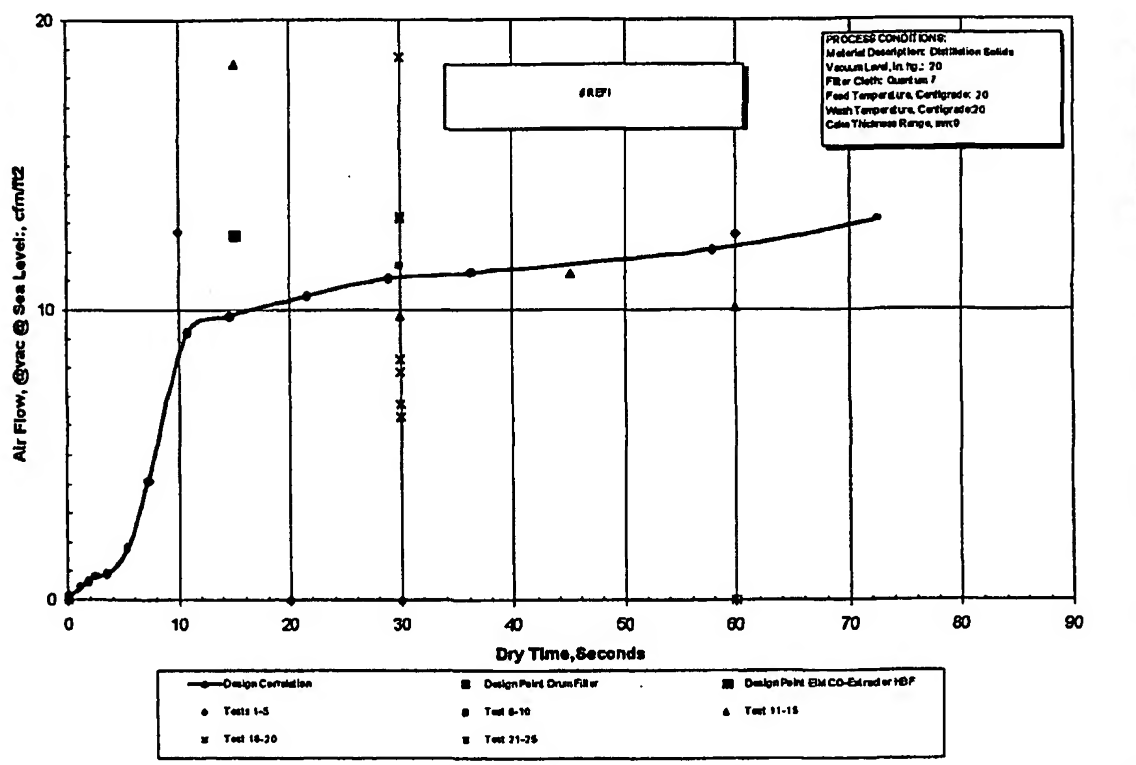
Θ_d = Dry Time, unit time

AIR FLOW

The air rate is shown with respect to the dry time in Figure 5. The air flow rate increases with time as the liquor in the filter cake is removed. Once the dry time is determined then the air rate can be calculated to size the vacuum pump for this application. The design point selected for this application is at the expected design cake thickness. The air rates listed in the figure for the specific filter type corrects the air flow rate during the dry period to the entire filter cycle time .

Figure 5

Air Rate vs θ_d



This factors in the selected dry time and the related filter functions of form, wash and cake discharge.

CAKE WASHING EFFECTIVENESS

Cake washing on a vacuum filter is accomplished by displacement of the mother liquor from the formed cake. Data are normally correlated by plotting the Fraction of solute remaining, R, in the cake that remains in solution with respect to the number of wash displacements, N. This is the volume of wash liquor applied/volume of final cake liquor. The relationships are shown in Equation 6 and Equation 7.

Equation 6

$$R = \frac{C_f - C_w}{C_l - C_w}$$

Where:

C_f = Concentration of Solute in final cake liquor

C_l = Concentration of Solute in Feed Liquor

C_w = Concentratioln of solute in wash liquor

Equation 7

$$N = \frac{V_w}{V_c}$$

Where:

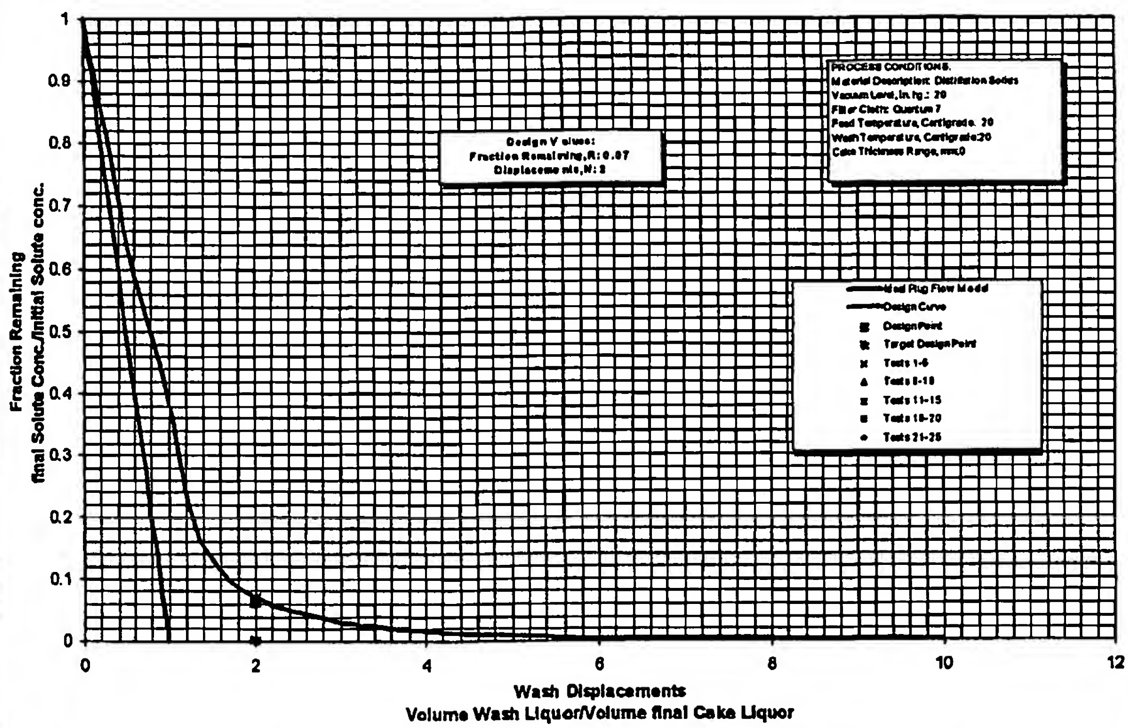
V_w = Volume of Wash Liquor

V_c = Volume of final Cake liquor (at end of dry period)

For purposes of calculation the moisture content is assumed to be the volume of the final cake liquor. The volume of the mother liquor can be calculated using the liquor density. With application of the wash water the solute concentration is reduced and the liquor concentration will change. Because the density of the final cake liquor is not measured for each test, using the moisture content simplifies the calculation and allows the use of water at a density of 1 g/cc. The test data are shown in Figure 6. The plug flow displacement is shown for reference and is used for calculation purposes in this evaluation.

Once the total wash volume exceeds 4-5 displacements, or Three stages of counter-current wash in this application, the removal of additional solute is negligible.

Figure 6
R vs N



RATE CALCULATIONS

Calculation of the filtration rate for vacuum filters is accomplished using Equation 8 and the function times determined from the various correlation's presented in the previous discussions.

Equation 8

$$FSFR = \frac{Wx60}{\Theta_{ct}}$$

Where:

FSFR = Full Scale Filtration Rate

60 = Conversion Factor, minute / hour

Θ_{ct} = Cycle Time, minutes

The cycle time for horizontal belt filters is the sum of the filter function times, as shown in Equation 9. The intermediate function times. θ_i ,would include intermediate dry periods, wash periods, steam dry periods, etc.

Equation 9
'Horizontal Belt Filter Cycle Time

$$\Theta_{ct} = \frac{\Theta_f + \Theta_i + \ldots + \Theta_d}{0.8}$$

Where:

Θ_{ct} = **Cycle Time, minutes**

Θ_f = **Form Time, minutes**

Θ_i = **Filter Function Time, minutes (any filter functions required)**

Θ_d = **Dry Time, minutes**

0.8 = Scale up factor

FILTER SIZING

Filter sizing is accomplished using the data correlation's and relationships described previously. Table 5 lists a summary of the sizing criteria, The filter constants from each the correlation's are selected. From these constant's the filter function times are calculated as previously discussed. A specific cake thickness is input into the data sheet and the rate is calculated for the specific condition and filter type.

Table 5
Filter Sizing Summary

		Process 100	Process 300
		EIMCO-Extractor HBF	EIMCO-Extractor HBF
Feed Rate, lbs dry solids/hr		129180	129180
Design Filter Area, ft²		5412	6111
Filter Cloth		QA7	QA7
Vacuum Level, in.Hg.		20.00	20.00
Air Rate, cfm/ft2 @ Vacuum @ Sea Level		3.26	2.89
Feed Concentration, wt%		20-25	20-25
Feed Temperature, °C		105	105
Wash Temperature, °C		60	60
Slurry pH		1-2	1-2
Design Cake Thickness, mm		25.00	25.00
Design Cake Weight, lbs/ft2		1.65	1.65
Design Cake Moisture, wt%		67.0%	67.0%
Fraction Remaining, R final conc/initial conc.		0.07	0.07
Wash Displacements, N volume wash/volume cake liquor		1.89	2.17
Number of Wash Stages		3	3
Design Influent Solute Concentration, wt%:		9.1	9.1
Solute Concentration Final Cake Liquor, wt%		<0.4	<0.4
Filtration Rate, lbs/hr/ft^2 (incl 0.8 Scale up)		24	21
1	Form Time, seconds	11	11
2	Wash Time, seconds	173	198
3	Dry Time, seconds	15	15
	Cycle Time, Minutes	4.15	4.68
Correlation Constants			
k_1	Cake Thickness ($W=k_1T$)	0.07	0.07
k_2	Form Time ($W=k_2T^{s-f}$)	0.50	0.50
k_3	Wash ($W/w=k_3$*Wash Time)	0.02	0.02
k_4	Dry (Dry Time/W)	8.39	9.39
s	Slope ($W=k_2T^s$)	0.50	0.50

Filter Press Dewatering

Dewatering tests using a filter press were performed using a small laboratory test unit. The hydrolazate material was relatively coarse and caused problems in attempting to fill the small laboratory filter. The coarse solids caused plugging in the feed pump. Tests were performed by screening out the + 16 mesh solids.

The hydrolazate material, representing the 100 and 300 1st stage process, filters very rapidly. A one inch cake forms at 10 psig within 10 seconds. The result is that a filter chamber is filled at very low pressure. Any increased pressure occurs near the center of the chamber with little compaction of the filter cake. The filter produces a cake containing 21 lbs. dry solids/ft3 of filter volume. The final cake solids are 30-35 wt%, depending on the effectiveness of an air blow to displace residual liquor. Compare to the vacuum filter at 33 wt% solids. In this application a recessed plate pressure filter will not produce a cake solids greater than the vacuum filter. A membrane plate filter would increase the solids slightly, to 35-40 wt%, but is limited due to the structure of the coarse solids that matte and prevent compression of the solids. The fine solids can be compressed with a membrane plate to produce 39-44 wt% solids.

The filtration time is a function of the pumping rate to the filter. Table 6 lists the basic feed and wash rates for this application. The sizing is based on four machines with about 86% availability for filtration, cake washing, discharge and cloth washing. The number of chambers can be changed to affect the availability number.

Counter-current washing on filter presses is NOT recommended. An intermediate surge tank would be required to hold the wash water between filter cycles. The volume would require 10,000-20,000 gal vessels, which could be provided. The main issue is that the filtrate liquid would need to be free of suspended solids. If process solids are in the wash liquid they will filter onto the back of the filter cloth. These can not be expected to be removed during the next filtration cycle such that eventually a portion of the filtrate drainage deck will become plugged with solids. This will affect the filtration and washing performance.

Wash temperature will need to be selected carefully. The filter plates selected for this application are high temp polypropylene plates. Although acceptable in higher temperatures, the plate material is susceptible to thermal shock. The plate temperature needs to change gradually and therefore the differential temperature between the process temperature and the wash temperature should be minimized.

Membrane plates were not evaluated on the hydrolazate material. The use of membranes will increase the solids concentration slightly 3-7 wt%, based on tests with the distillate solids.

An automated filter press is recommended to provide less operator attention. The automated filter will require a cloth shaking device and a flood/cloth wash to remove residual cake from the chambers.

Because of the low cake density there is a potential that some of the cakes may leave pieces in the filter chambers. For conventional filter presses this may require an operator present during discharge and will result in increased discharge times to move each plate individually. The result is a short processing cycle 10-15 minutes and a long discharge and cloth wash cycle of 45-50 minutes. Cloth washing may be required and the sizing of conventional filters assumes one wash cycle/day for an average of 10 minutes per cycle. The automated filter will

wash on each cycle but will only require 2 minutes maximum/cycle. The flood wash used with the automated filter will remove residual pieces of cake that may hang up. The result is that some additional processing capacity will be required.

The cloth wash quantity will be large volume and short duration. The water will become contaminated with the residue solids. For design purposes a clarifier should be provided to collect this water, clarify it and provide the source for the next flood wash cycle. Based on a flow of about 24,000-30,000 gal/hr (500 gpm) a 50-ft diameter clarifier is suggested.

Table 6
Filter Press Sizing Summary

	2 x 2 meter plates
Feed Rate, lbs. slurry/hr	494,942
Solids Concentration, wt%	26%
Solids Rate, lbs./hr	129,180
Dilution Concentration, wt%	21%
Cake Density, lbs/ft^3	21
Filter Volume required, ft^3/hr	6151
Filtration Time, minutes	6
Wash Time, minutes	6
Air Blow Time, minutes	2
Discharge Time, minutes	3
Cloth/Flood Wash, minutes	2
Cycle Time, minutes	19
Cycles/hour	3.16
Feed Rate, gpm (average)	
Air Req'd, scfm/ft2	1.00
Feed Rate, gpm/filter (average filtration time))	982
Wash Rate, gpm/filter (During Wash Period)	963
Filter Volume Required, ft^3/cycle	1948
Volume, ft^3/chamber	5.65
Number of Chambers	345
Number of Chambers/Filter	100
Number of Filters	3.45
Filter Availability (Based on 4 units)	86%

CONCLUSIONS AND RECOMMENDATIONS

Based on the filtration tests results from the hydrolazate material the use of horizontal belt filter is recommended for dewatering and washing the solids. The requested design is for 5-6 counter-current wash stages/filter. Generally with a 2-displacement wash the use of 3-4 stages is the maximum number that is required to effectively remove the solute. It is recommended that the evaluation include the reduced number of wash stages because of the savings in the number of filters required to process the solids. With the requested number of stages the 100 Process will require six (6) 145 m^2 filters, and the 300 Process will require seven (7) of these units. If the number of wash stages is reduced to three (3), then the number of filters will be four (4) for both processes.

Filter presses may be used in this application. The recommended design would be with an automated design to insure cake discharge. This is necessary to reduce the equipment size and reduce operating labor. Standard recessed or membrane plate filter presses will require operator attention to insure that cake discharge occurs. With the size of the filters required 2 x 2 meter or larger with 125-150 plates/filter a total of 8 filter would be required. With the expected discharge time a minimum of one operator would be required for every two filters. With Automated filters one operator should be able to handle the entire filter system and four filters would be required. The automated option will require a thickener to clarify the cloth wash/flood wash water.

CHEMICAL ANALYSIS & TESTING

Analytical Report Baker Hughes

Analysis No.	Page
2000-050	1 of 1

Project Title: Harris Group Dewatering Test Work Samples

NREL In-House	Current Subcontractor	CRADA	Other
[X]	[ ]	[ ]	[X]

Name of Project Contact Person:	Andy Aden	Date Samples Delivered:	05-04-00
NREL Notebook:	NB# 2275, pp 062-063	Date Work Completed:	06-12-00
Samples Number and Type:	13 solids and 8 liquids	Actual Hours Spent:	14
Summary of work requested:	Analysis of samples for total solids content, glucose, xylose, and acetic-acid.	Proposed Approach:	Analyzed in-house according to NREL Laboratory Analytical Procedures.

Work required: Sample Prep [X] Acid Digest [ ] HPLC [X] YSI [ ] GC [ ] CHN [ ] Other: [ ]

Results and Comments

	[X] mg/ml			[X] % Dry Weight
Sample	G	X	AA	TS
1 Beltpress Test D-1 filter cake	nd	nd	nd	27.32
2 Beltpress Test D-2 filter cake	nd	nd	nd	26.80
3 Beltpress Test D-2F filter cake	nd	nd	nd	34.16
4 Beltpress Test D-3 filter cake	nd	nd	nd	24.74
5 Beltpress Test D-4 filter cake	nd	nd	nd	25.33
6 Beltpress Test D-4E filter cake	nd	nd	nd	28.39
7 Pressure filter test P-3 filter cake	nd	nd	nd	49.61
8 Pressure filter test P-4 filter cake	nd	nd	nd	42.09
9 Pressure filter test P-5 filter cake	nd	nd	nd	35.59
10 Pressure filter test P-6 filter cake	nd	trace	nd	37.26
11 Centrifuge test 1 cake, distillate	nd	trace	nd	23.10
12 Centrifuge test 2 cake, distillate	trace	trace	nd	22.55
13 Centrifuge test 3 cake, distillate	0.19	trace	nd	23.08
14 Centrifuge test 1 centrate, distillate	nd	0.48	0.83	2.85
15 Centrifuge test 2 centrate, distillate	nd	0.52	0.94	3.17
16 Centrifuge test 3 centrate, distillate	nd	0.57	1.29	3.18
17 Beltpress test D-1 gravity drain liquor	nd	nd	3.57	2.48
18 Beltpress test D-2 gravity drain liquor	nd	nd	2.53	2.35
19 Beltpress test D-4 gravity drain liquor	nd	0.32	2.08	2.40
20 BP pressure composite	nd	0.09	5.02	3.52
21 P-6, pressure filter test 6 filtrate	0.09	0.06	2.03	2.67

A=arabinose; AA=acetic acid; CEL=cellobiose; ET=ethanol; FA=formic acid; FL=furfural; G=glucose; GA=galactose; GLY=glycerol; HMF=5-hydroxymethyl-2-furaldehyde; LA=levulinic acid; LAC=lactic acid; LAS=acid soluble lignin; M=mannose; n/a=not applicable; nd=not detected; nr=not requested; SUC=succinic acid; TS=total solids; X=xylose; XYL=xylitol

Name(s) of CAT Staff Working on Project:	Reviewed by:
Ray Ruiz	

APPENDIX C
BLACK CLAWSON

H

HARRIS GROUP INC.
PROJECT NO: 99-10600
REVISION B Mar-06-01

NATIONAL RENEWABLE ENERGY LABORATORY
GOLDEN, COLORADO
HORIZONTAL BELT FILTER - PROCESS 100 - LIQUID SOLID SEPARATION

MECHANICAL EQUIPMENT LIST

EQUIP # REV MOTOR #	DESCRIPTION	VENDOR P.O. ISSUED	SIZE CAPACITY HEAD	GEAR RATIO	EQUIP STATUS	HORSEPOWER RPM VOLTS	ENCLOSURE FRAME MODEL NO.	REMARKS
	MIX FEED TANK #1		11' DIA. X 11' H 7500 GAL					FRP CONSTRUCTION
	MIX FEED TANK #2		11' DIA. X 11' H 7500 GAL					FRP CONSTRUCTION
	MIX FEED TANK #3		11' DIA. X 11' H 7500 GAL					FRP CONSTRUCTION
	MIX FEED TANK AGITATOR #1					25 HP		
	MIX FEED TANK AGITATOR #2					25 HP		
	MIX FEED TANK AGITATOR #3					25 HP		
	HORIZONTAL BELT FILTER #1	BLACK CLAWSON						
	HORIZONTAL BELT FILTER #2	BLACK CLAWSON						
	HORIZONTAL BELT FILTER #3	BLACK CLAWSON						
	FEED PUMP #1	GOULDS	MOD 3196 3X4-10 280 GPM 60 FT TDH			7.5 HP 1800 RPM		316 SS CONSTRUCTION
	FEED PUMP #2	GOULDS	MOD 3196 3X4-10 280 GPM 60 FT TDH			7.5 HP 1800 RPM		316 SS CONSTRUCTION
	FEED PUMP #3	GOULDS	MOD 3196 3X4-10 280 GPM 60 FT TDH			7.5 HP 1800 RPM		316 SS CONSTRUCTION
	FILTRATE RETURN PUMP #1	GOULDS	MOD 3196 3X4-10H 450 GPM 60 FT TDH			10 HP 1800 RPM		316 SS CONSTRUCTION
	FILTRATE RETURN PUMP #2	GOULDS	MOD 3196 3X4-10H 450 GPM 60 FT TDH			10 HP 1800 RPM		316 SS CONSTRUCTION
	FILTRATE RETURN PUMP #3	GOULDS	MOD 3196 3X4-10H 450 GPM 60 FT TDH			10 HP 1800 RPM		316 SS CONSTRUCTION
	FILTRATE PUMP #1	GOULDS	MOD 3196 3X4-10H 400 GPM 60 FT TDH			10 HP 1800 RPM		316 SS CONSTRUCTION
	FILTRATE PUMP #2	GOULDS	MOD 3196 3X4-10H 400 GPM 60 FT TDH			10 HP 1800 RPM		316 SS CONSTRUCTION
	FILTRATE PUMP #3	GOULDS	MOD 3196 3X4-10H 400 GPM 60 FT TDH			10 HP 1800 RPM		316 SS CONSTRUCTION
	WASH PUMP #1A THRU E	GOULDS	MOD 3196 3X4-10H 350 GPM 60 FT TDH			10 HP 1800 RPM		316 SS CONSTRUCTION 5 PUMPS
	WASH PUMP #2A THRU E	GOULDS	MOD 3196 3X4-10H 350 GPM 60 FT TDH			10 HP 1800 RPM		316 SS CONSTRUCTION 5 PUMPS
	WASH PUMP #3A THRU E	GOULDS	MOD 3196 3X4-10H 350 GPM 60 FT TDH			10 HP 1800 RPM		316 SS CONSTRUCTION 5 PUMPS

[redacted]

THERMO BLACK CLAWSON INC.
A Thermo Fibertek company

April 7, 2000

Mr. Lynn Montague
Harris Group Inc.
1000 Denny Way
Suite 800
Seattle, WA 98109-5338

SUBJECT: Thermo Black Clawson Inc. Quotation No.5212

Dear Mr. Montague,

Please find enclosed the Thermo Black Clawson's Quotation No.5212 for a 1500 BDST/D fermented biomass washing equipment. Attached also are the mass balances for Process 100 and Process 300 for your review.

The Process 100 would require three type "D" 8m x 20m 5 stage Chemi-Washers to process 1,500 BDST of the fermented biomass. The washers would be run with a dilution factor of 1.5, which translates into 940 gallons of wash water per BDST of the fermented biomass. The washed biomass would be discharged at a consistency of about 26%. As requested, acetic acid concentration in the filtrate from the washed biomass would be no higher than 1.7 g/l.

The Process 300 would require four type "E" 10m x 22m 6 stage Chemi-Washers to process the same as above amount of the fermented biomass. The washers would be run with a 2.5 dilution factor, which corresponds to 1,105 gallons of wash water per BDST of the fermented biomass. As requested, the washing efficiency for glucose would be no less than 95%.

We are confident that the equipment in this quotation will provide the most cost efficient process solution for the project.

We look forward to working with Harris Group Inc. on this project. Should you have any questions, please contact the writer at (513) 420-8383 or Mr. Guillermo Dietrich at (513) 420-8385.

Very Truly Yours,

THERMO BLACK CLAWSON INC.

R Szopinski

Ryszard Szopinski
Product Manger

cc: Mike Stephens
Capital Sales
CPG

Cover letter to Montague.doc

THERMO BLACK CLAWSON INC.
A Thermo Fibertek company
Middletown, Ohio USA

QUOTATION No. 5212

ONE BLACK CLAWSON TYPE "D" CHEMI-WASHER®

533.3 BDST/D Fermented Biomass

FOR

HARRIS GROUP INC.
SEATTLE, WA

FROM

THERMO BLACK CLAWSON INC.

CHEMICAL PULPING GROUP

MIDDLETOWN, OHIO USA

PERFORMANCE DATA

The pulp to be washed is defined as follows:

Raw Material	Fermented Biomass
Headbox Consistency	12.0% minimum
Inlet pulp temperature	100°C
Drainage Characteristics	Tested by Thermo Black Clawson Inc.

For this pulp the Chemi-Washer will operate under the following conditions:

Design Tonnage	533.3 BDST/D
Suction Area	160 m^2
Suction Width	8 m
Suction Length	20 m
Wire Width	8.15 m
Basis Weight	0.52 lbs/ft^2
Speed	55 ft/min
Wash Water Temperature	60°C minimum
Dilution Factor	1.5
Wash Stages	5
Vacuum	0.6-1.0 m H_2O
Hand of Machine	Right*

*NOTE: When standing in the tending side aisle, and facing the machine, the stock will travel from left to right.

THERMO BLACK CLAWSON INC.

A Thermo Fibertek company
Middletown, Ohio USA

QUOTATION No. 5212

ONE BLACK CLAWSON TYPE "E" CHEMI-WASHER®

400 BDST/D Fermented Biomass

FOR

HARRIS GROUP INC.

SEATTLE, WA

FROM

THERMO BLACK CLAWSON INC.

CHEMICAL PULPING GROUP

MIDDLETOWN, OHIO USA

PERFORMANCE DATA

The pulp to be washed is defined as follows:

Raw Material	Fermented Biomass
Headbox Consistency	12.0% minimum
Inlet pulp temperature	100°C
Drainage Characteristics	Tested by Thermo Black Clawson Inc.

For this pulp the Chemi-Washer will operate under the following conditions:

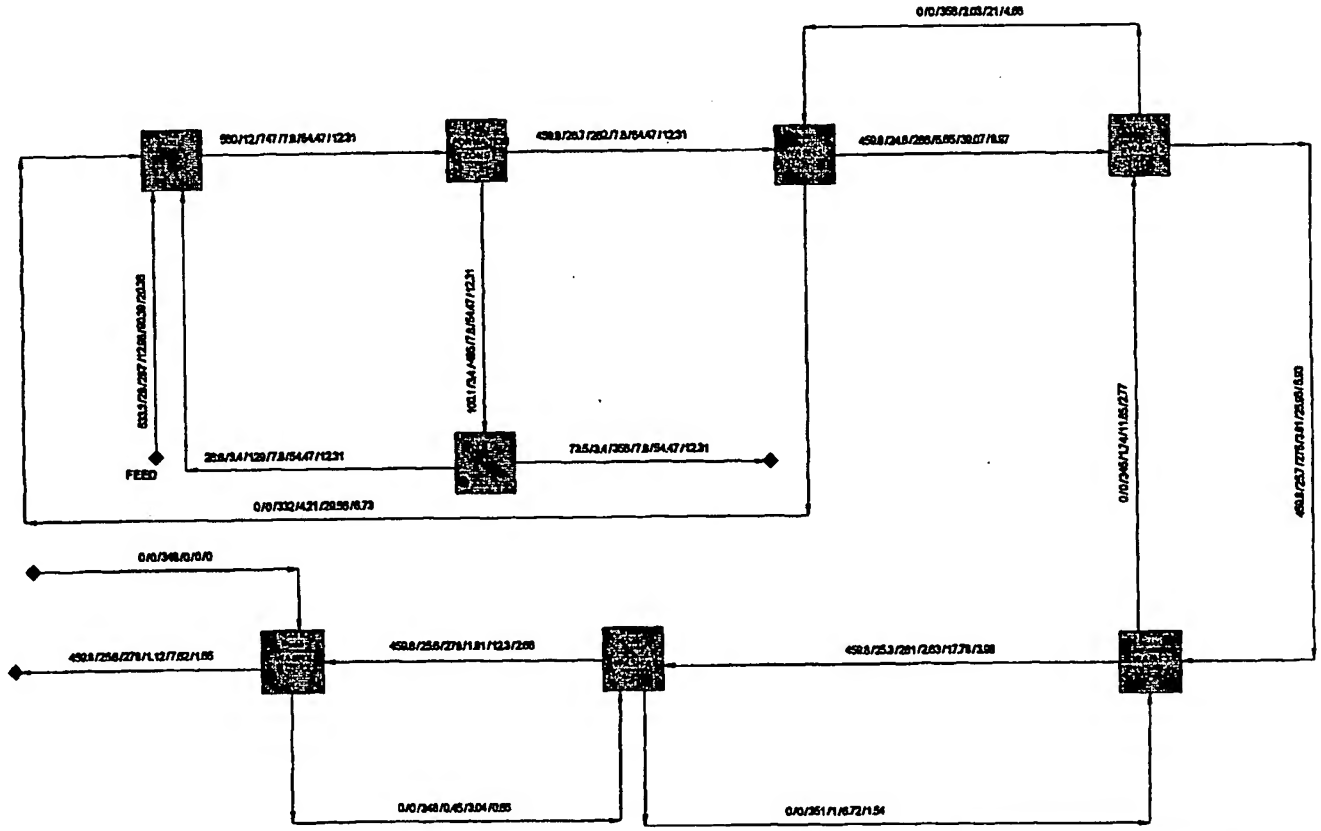
Design Tonnage	400 BDST/D
Suction Area	220 m^2
Suction Width	10 m
Suction Length	22m
Wire Width	10.15 m
Basis Weight	0.52 lbs/ft^2
Speed	33 ft/min
Wash Water Temperature	60°C minimum
Dilution Factor	2.5
Wash Stages	6
Vacuum	0.6-1.0 m H_2O
Hand of Machine	Right*

*NOTE: When standing in the tending side aisle, and facing the machine, the stock will travel from left to right.

HARRIS GROUP INC.

SEATTLE, WA

PROCESS 100 533.3 BDST/DAY

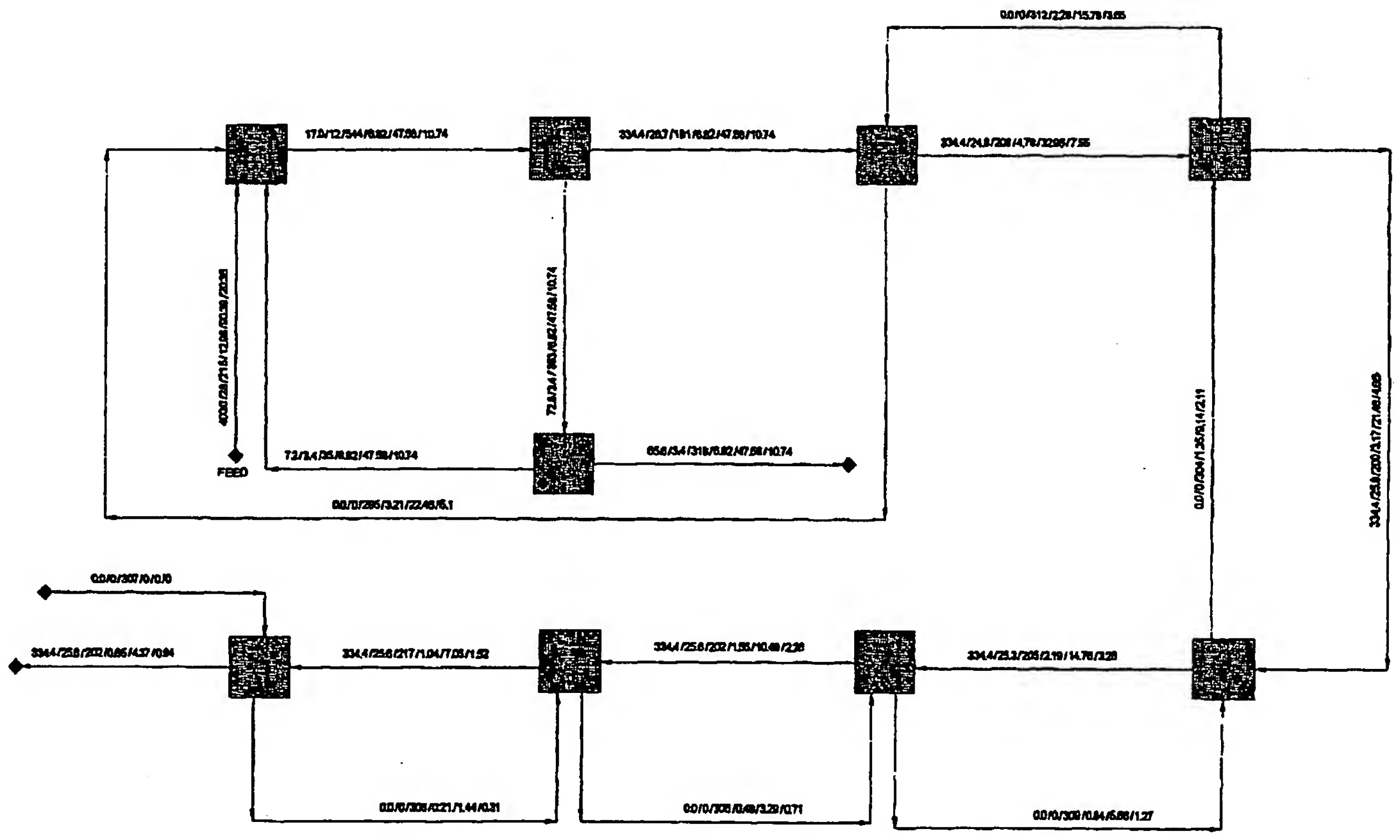


[redacted]

HARRIS GROUP INC.

SEATTLE, WA

PROCESS 300 400 BDST/DAY



[redacted]

THERMO BLACK CLAWSON INC.

A Thermo Fibertek company

Technical Report

Recycled Fiber • Chemical Pulping • Stock Preparation

THERMO BLACK CLAWSON INC.

A Thermo Fibertek company

Technology Center - Middletown, Ohio

April 6, 2000

Report Number: 41,031

Counter-Current Washing Trials on Bagasse Biomass

For:

Harris Group Inc.
Seattle, WA

By:

Guillermo Dietrich-Velázquez

Thermo Black Clawson Distribution

Max Caldwell / R&D Files
Percy Brooks / D. E. Chupka / R&D Files
Michael A. Sieron / R&D Files
Mgrs./Applications Engineers / R&D Files
J. C. Kerr / Business Development
Kurt Kobelt
John Cannon

Customer Distribution

John C. Lukas
Lynn Montague

THERMO BLACK CLAWSON INC.

A Thermo Fibertek company

Report Number: 41,031 | Date: **April 6, 2000**

Counter-Current Washing Trials on Bagasse Biomass

SUMMARY

The objective of this trial was to evaluate the washing efficiency of a 4-stage, counter-current washing process, on bagasse biomass using a drainage tester.

The consistency of the as-received, fermented bagasse solids (FSAR) was 28.1%; it's texture is very similar to a paper mill's sludge. The liquid portion of the FSAR is a solution of sugars, acetic acid and other organic compounds in an aqueous, diluted acidic media having a pH of 1.6. It was determined that each pound of FSAR required ~1.5 pounds of water or dilution liquor to make it pumpable

Seven different washing tests were performed. These tests simulated one or 4 washing stages, with fresh water additions of 0.59, 0.86 or 1.11 pounds per pound of FSAR. The fresh water temperature was 117 or 145°F (47 or 63°C). Each test was run by triplicate.

Samples of each individual washing stage filtrate, washed solids, composite samples of fresh water, shower liquors and FSAR were collected and sent to an independent laboratory for analysis of acetic acid and sugars. From the analytical data, the washing efficiency for these components was determined.

For one process (Process 100, tests 2 and 5) the desired acetic acid content in the washed biomass should be a maximum of 3.3 g/Kg of liquor at 27% consistency, with a desirable level of 1.65 g/Kg liquor. This is equivalent to a removal efficiency of 84% to 92%.

For another process (Process 300, tests 3, 4, 6, 7 and 8) the desired sugar removal efficiency should be at least 95%, with 98% being preferred.

The production rate for either process is close to1600 oven-dry short tons per day.

It was found that the drainage characteristics of the FSAR are poor, so a large drainage table will be required.

It was also found that the addition of 0.59 lb water / lb of FSAR leads to a negative Dilution Factor giving a very low washing efficiency. It will be economically prohibitive to build a machine with 8 or more washing stages to wash a fraction of the production required at such operating conditions.

The considerable amount of fines present in the FSAR led to a solids loss in the Formation Zone of approximately 18%. If the excess filtrate from the Forming Zone is

CHEMIWASHER DRAINAGE TEST DATA SHEET

Company	Harris Group Inc.	%C as received	28.1	Total Solids in liquor, %	
Location	Seattle, WA	DS in pulp's liquor		Suspended Solids in Dil. Liq., %	1.45
Pulp Type	Fermented Bagasse	Pulp's CSF, ml		Sp. Grav. Dilution Liqour:	1.02 @ 80 ° C
Kappa Number		Additional Sample Data	Test # 4 . 1.11 lb Fresh Water @ 64 ° C / lb FSAR		

		FEED SLURRY			FORMING ZONE		1ST STAGE		2ND STAGE		3RD STAGE	
Run No.	Date	Weight, grams	Consist. %	Temp. °C	Temp. °C	Drain Time, Secs.	Temp. °C	Drain Time, Secs.	Temp. °C	Drain Time, Secs.	Temp. °C	Drain Time, Secs.
1	1/17/00	168	11.2	100	98	6.4	64	7.6	--	--	--	--
2	1/17/00	168	11.2	98	98	6.9	64	8.3	--	--	--	--
3	1/17/00	168	11.2	100		7.6	64	8.1	--	--	--	--
AVERAGE →						7.0		8.0				

4TH STAGE		5TH STAGE	
Temp. °C	Drain Time, Secs.	Temp. °C	Drain Time, Secs.
--	--	--	--
--	--	--	--
--	--	--	--

←AVERAGE

REMARKS:

Filtrate Collected: 85.2 g, 78.1 g, 80.3 g. Average = 81.2 g

Discharge Consistency: 26.8%, 26.9%, 26.7%. Average = 26.8%

ZONE VACUUMS (inches of water)			
F.Z.	8	4th Stage	
1st Stage	30		
2nd Stage			
3rd Stage			

WASH VOLUME, ml	
1st Stage	75
2nd Stage	
3rd Stage	
4th Stage	

Tested by	GDV
Location	TBC Tech. Ctr.

Project # 41,031

CHEMIWASHER DRAINAGE TEST DATA SHEET

Company	Harris Group Inc.	%C as received	28.1	Total Solids in liquor, %	
Location	Seattle, WA	DS in pulp's liquor		Suspended Solids in Dil. Liq., %	1.45
Pulp Type	Fermented Bagasse	Pulp's CSF, ml		Sp. Grav. Dilution Liqour:	1.02 @ 80 ° C
Kappa Number		Additional Sample Data	Test # 5.	0.59 lb Fresh Water @ 47°C / lb FSAR	

		FEED SLURRY			FORMING ZONE		1ST STAGE		2ND STAGE		3RD STAGE	
Run No.	Date	Weight, grams	Consist. %	Temp. °C	Temp. °C	Drain Time, Secs.	Temp. °C	Drain Time, Secs.	Temp. °C	Drain Time, Secs.	Temp. °C	Drain Time, Secs.
1	1/17/00	168	11.2	98	96	7.7	56	5.8	53	5.5	50	4.7
2	1/17/00	168	11.2	100	96	7.3	56	5.6	53	5.3	50	4.6
3	1/17/00	168	11.2	99	97	7.9	58	5.6	53	5.7	50	5.0
AVERAGE →						7.6		5.7		5.5		4.8

4TH STAGE		5TH STAGE	
Temp. °C	Drain Time, Secs.	Temp. °C	Drain Time, Secs.
48	4.2	—	—
48	4.7	—	—
48	4.6	—	—
	4.5		

← AVERAGE

REMARKS:

Filtrate Collected, average

1st Stage: 36.8 g   2nd Stage: 38.6 g   3rd Stage: 43.2 g   4th Stage: 43.7 g

Discharge Consistency: 25.4%, 25.8%, 26.0%. Average = 25.7%

ZONE VACUUMS (inches of water)			
F.Z.	7	4th Stage	30
1st Stage	13		
2nd Stage	18		
3rd Stage	24		

WASH VOLUME, ml	
1st Stage	40
2nd Stage	40
3rd Stage	40
4th Stage	40

Tested by GDV
Location TBC Tech. Ctr.

Project # 41,031

CHEMIWASHER DRAINAGE TEST DATA SHEET

Company	Harris Group Inc.	%C as received	28.1	Total Solids in liquor, %	
Location	Seattle, WA	DS in pulp's liquor		Suspended Solids in Dil. Liq., %	1.45
Pulp Type	Fermented Bagasse	Pulp's CSF, ml		Sp. Grav. Dilution Liqour:	1.02 @ 80 ° C
Kappa Number		Additional Sample Data	Test # 6.	0.86 lb Fresh Water @ 64°C / lb FSAR	

		FEED SLURRY			FORMING ZONE		1ST STAGE		2ND STAGE		3RD STAGE	
Run No.	Date	Weight, grams	Consist. %	Temp. °C	Temp. °C	Drain Time, Secs.	Temp. °C	Drain Time, Secs.	Temp. °C	Drain Time, Secs.	Temp. °C	Drain Time, Secs.
1	1/14/00	168	11.2	100	100	7.1	70	6.0	67	6.0	65	5.0
2	1/14/00	168	11.2	100	96	6.6	70	6.3	68	5.4	67	4.9
3	1/14/00	168	11.2	100	98	7.1	71	5.9	68	5.5	66	5.3
AVERAGE →						6.9		6.1		5.6		5.1

4TH STAGE		5TH STAGE	
Temp. °C	Drain Time, Secs.	Temp. °C	Drain Time, Secs.
64	5.0	—	—
64	4.6	—	—
64	4.4	—	—
	4.7		

←AVERAGE

REMARKS:
Filtrate Collected, Average
1st Stage: 53.6 g 2nd Stage: 56.7 g 3rd Stage: 62.1 g 4th Stage: 60.9 g

Discharge Consistency: 25.7%, 26.4%, 26.1%. Average = 26.1%

ZONE VACUUMS (inches of water)			
F.Z.	7	4th Stage	30
1st Stage	13		
2nd Stage	18		
3rd Stage	24		

WASH VOLUME, ml	
1st Stage	58
2nd Stage	58
3rd Stage	58
4th Stage	58

Tested by	GDV
Location	TBC Tech. Ctr.

Project # 41,031

CHEMIWASHER DRAINAGE TEST DATA SHEET

Company	Harris Group Inc.	%C as received	28.1	Total Solids in liquor, %	
Location	Seattle, WA	DS in pulp's liquor		Dissolved Solids in liquor, %	
Pulp Type	Fermented Bagasse	Pulp's CSF, ml		Sp. Grav. Dilution Liqour:	1.02 @ 80 ° C
Kappa Number		Additional Sample Data	Test # 7.	1.11 lb Fresh Water @ 64°C / lb FSAR	

		FEED SLURRY			FORMING ZONE		1ST STAGE		2ND STAGE		3RD STAGE	
Run No.	Date	Weight, Grams	Consist. %	Temp. °C	Temp. °C	Drain Time, Secs.	Temp. °C	Drain Time, Secs.	Temp. °C	Drain Time, Secs.	Temp. °C	Drain Time, Secs.
1	1/13/00	168	11.2	100	99	7.7	71	7.5	68	7.5	66	5.4
2	1/13/00	168	11.2	100	98	7.2	69	7.1	66	6.5	65	6.0
3	1/13/00	168	11.2	100	95	8.0	72	7.0	69	7.0	64	6.0
AVERAGE →						7.6		7.2		7.0		5.8

4TH STAGE		5TH STAGE	
Temp. °C	Drain Time, Secs.	Temp. °C	Drain Time, Secs.
64	6.1	---	---
64	6.0	---	---
64	6.2	---	---

←AVERAGE

REMARKS:
Filtrate Collected, average:
1st Stage: 71.4 g 2nd Stage: 76.4 g 3rd Stage: 73.8 g 4th Stage: 75.7 g

Discharge Consistency: 26.6%, 27.7%, 26.1%. Average = 26.8%

ZONE VACUUMS (inches of water)			
F.Z.	7	4th Stage	30
1st Stage	13		
2nd Stage	18		
3rd Stage	24		

WASH VOLUME, ml	
1st Stage	75
2nd Stage	75
3rd Stage	75
4th Stage	75

Tested by	GDV
Location	TBC Tech. Ctr.

Project # 41,031

CHEMIWASHER DRAINAGE TEST DATA SHEET

Company	Harris Group Inc.	%C as received	28.1	Total Solids in liquor, %	
Location	Seattle, WA	DS in pulp's liquor		Dissolved Solids in liquor, %	
Pulp Type	Fermented Bagasse	Pulp's CSF, ml		Sp. Grav. Dilution Liqour:	1.02 @ 80 ° C
Kappa Number		Additional Sample Data	Test # 8 .	0.59 lb Fresh Water @ 64°C / lb FSAR	

		FEED SLURRY			FORMING ZONE		1ST STAGE		2ND STAGE		3RD STAGE	
Run No.	Date	Weight, Grams	Consist. %	Temp. °C	Temp. °C	Drain Time, Secs.	Temp. °C	Drain Time, Secs.	Temp. °C	Drain Time, Secs.	Temp. °C	Drain Time, Secs.
1	1/14/00	168	11.2	100	97	7.1	70	5.6	67	5.3	65	4.1
2	1/14/00	168	11.2	100	96	6.4	70	5.0	66	4.9	66	4.0
3	1/14/00	168	11.2	100	96	6.4	71	5.4	66	5.0	64	4.1
AVERAGE →						6.6						4.1

4th STAGE		5TH STAGE	
Temp. °C	Drain Time, Secs.	Temp. °C	Drain Time, Secs.
64	3.8	—	—
63	3.3	—	—
64	3.6	—	—
	3.6		

←AVERAGE

REMARKS:
Filtrate Collected, Average
1st Stage: 36.5 g 2nd Stage: 42.9g 3rd Stage: 40.6 g 4th Stage: 42.6g

Discharge Consistency: 26.3%, 25.7%, 26.5%. Average = 26.2%

ZONE VACUUMS (inches of water)			
F.Z.	7	4th Stage	30
1st Stage	13		
2nd Stage	18		
3rd Stage	24		

WASH VOLUME, ml	
1st Stage	40
2nd Stage	40
3rd Stage	40
4th Stage	40

Tested by	GDV
Location	TBC Tech. Ctr.

Project # 41,031

CHEMICAL ANALYSIS & TESTING

Analytical Report

2000-0328 | 1 of 1

Project Title:	BlackClawson Solids

NREL In-House [X] | Current Subcontractor [ ] | CRADA [ ] | Other [ ]

Name of Project Contact Person:	Mark Ruth	Date Samples Delivered:	01/27/2000
NREL Notebook:	NB# 2275, pp 048-051	Date Work Completed:	02/04/2000
Sample Number and Type:	21 washed solids and 1 "biomass as received" solid	Actual Hours Spent:	20
Summary of work requested:	Analysis of solids for total solids content and for entrained glucose, xylose, and acetic-acid.	Procedures Approach:	Analyzed in-house according to NREL Laboratory Analytical Procedures.

Work required: Sample Prep [ ] | Acid Digest [ ] | HPLC [X] | YSI [ ] | GC [ ] | CHN [ ] | Other: [ ]

Results and Comments

	Sample	[X] mg/ml G	X	AA			[X] % Dry Weight TS
1	Washed Solids 1 stage Test #2 Run #1	3.49	23.55	5.73			31.58
2	Washed Solids 1 stage Test #2 Run #2	3.40	23.50	6.19			32.00
3	Washed Solids 1 stage Test #2 Run #3	3.79	26.31	6.92			31.58
4	Washed Solids 1 stage Test #3 Run #1	4.10	27.67	7.10			38.76
5	Washed Solids 1 stage Test #3 Run #2	2.42	16.42	4.31			29.84
6	Washed Solids 1 stage Test #3 Run #3	2.27	15.40	3.96			29.75
7	Washed Solids 1 stage Test #4 Run #1	2.40	16.35	4.02			30.42
8	Washed Solids 1 stage Test #4 Run #2	2.18	14.75	3.71			30.73
9	Washed Solids 1 stage Test #4 Run #3	1.88	12.50	3.13			30.77
10	Washed Pulp 4 stage Test #3 Run #1	0.76	4.75	1.21			28.11
11	Washed Solids 4 stage Test #3 Run #2	1.08	7.18	1.80			29.38
12	Washed Pulp 4 stage Test #9 Run #3	0.94	6.05	1.50			27.39
13	Washed Solids 4 stage CC washing Test #3 Run #1	0.64	3.89	0.98			27.84
14	Washed Solids 4 stage CC washing Test #3 Run #2	1.06	6.90	1.61			29.47
15	Washed Solids Test #5 Run #3	1.05	6.87	1.60			28.27
16	Washed Solids 4 stage countercurrent washing Test #7 Run #1	0.64	3.77	0.87			28.18
17	Washed Solids 4 stage washing 1.1 lb H2O / lb solids as received Test #7 Run #2	0.77	4.71	1.07			29.11
18	Washed Solids Test #7 Run #3	0.86	5.45	1.24			28.39
19	Washed Solids 4 stage Test #8 Run #1	1.54	10.54	2.52			29.39
20	Washed Solids 4 stage Test #8 Run #2	1.17	7.68	1.91			29.02
21	Washed Solids 4 stage Test #8 Run #3	1.30	8.71	2.06			28.39
22	*Biomass as received*	12.98	80.39	20.36			38.13
23							
24							

A=arabinose; AA=acetic acid; CEL=cellobiose; ET=ethanol; FA=formic acid; FL=furfural; G=glucose; GA=galactose; GLY=glycerol; HMF=5-hydroxymethyl-2-furaldehyde; LA=levulinic acid; LAC=lactic acid; LAS=acid soluble lignin; M=mannose; n/a=not applicable; nd=not detected; nr=not requested; SUC=succinic acid; TS=total solids; X=xylose; XYL=xylitol

Name(s) of CAT Staff Working on Project:	Reviewed by:
Ray Ruiz	

CHEMICAL ANALYSIS & TESTING

Analytical Report

Project Title:	BlackClawson Liquids

NREL In-House [X] Current Subcontractor [ ] CRADA [ ] Other [ ]

Name of Project Contact Person:	Mark Ruth	Date Samples Delivered:	01/27/2000
NREL Notebook:	NBB 2275, pp 048-051	Date Work Completed:	02/04/2000
Samples Number and Type:	63 liquor samples	Actual Hours Spent:	12
Summary of Work requested:	Analysis of liquids for total solids content and for free soluble glucose, xylose, and acetic-acid.	Procedures Applied:	Analyzed in-house according to NREL Laboratory Analytical Procedures.

Work required: Sample Prep [ ] Acid Digest [ ] HPLC [X] YSI [ ] GC [ ] CHN [ ] Other: [ ]

Results and Comments

Sample	[X] mg/ml G	X	AA		[X] % Dry Weight	TS
1 Forming Zone Filtrate	10.95	76.44	18.09			nr
2 Dilution Liquor	9.34	65.31	17.18			nr
3 First Stage Shower	3.77	26.28	6.82			nr
4 Second Stage Shower	1.62	11.06	2.93			nr
5 Third Stage Shower	0.76	4.98	1.34			nr
6 Fresh Water (4th Stage Shower)	nd	nd	nd			nr
7 Test #2 Run #1 1st Stage Filtrate	4.75	32.82	8.01			5.23
8 Test #2 Run #2 1st Stage Filtrate	5.36	38.00	9.67			5.86
9 Test #2 Run #3 1st Stage Filtrate	5.79	40.66	10.50			6.22
10 Test #3 Run #1 1st Stage Filtrate	4.19	28.79	7.16			4.47
11 Test #3 Run #2 1st Stage Filtrate	4.50	31.88	8.06			4.99
12 Test #3 Run #3 1st Stage Filtrate	4.93	35.06	8.99			5.37
13 Test #4 Run #1 1st Stage Filtrate	5.09	36.12	8.92			5.56
14 Test #4 Run #2 1st Stage Filtrate	5.25	37.35	9.64			5.73
15 Test #4 Run #3 1st Stage Filtrate	3.99	28.23	7.42			4.41
16 Test #5 Run #1 1st Stage Filtrate	6.17	42.32	10.52			6.58
17 Test #5 Run #1 2nd Stage Filtrate	5.75	39.63	9.86			6.03
18 Test #5 Run #1 3rd Stage Filtrate	3.32	22.92	5.86			3.58
19 Test #5 Run #1 4th Stage Filtrate	1.50	10.09	2.74			1.70
20 Test #5 Run #2 1st Stage Filtrate	5.89	41.38	10.53			6.37
21 Test #5 Run #2 2nd Stage Filtrate	5.19	36.63	9.35			5.59
22 Test #5 Run #2 3rd Stage Filtrate	2.97	20.89	5.33			3.29
23 Test #5 Run #2 4th Stage Filtrate	1.48	10.22	2.82			1.78
24 Test #5 Run #3 1st Stage Filtrate	5.28	36.90	9.17			5.71

A=arabinose; AA=acetic acid; CEL=cellobiose; ET=ethanol; FA=formic acid; FL=furfural; G=glucose; GA=galactose; GLY=glycerol; HMF=5-hydroxymethyl-2-furaldehyde; LA=levulinic acid; LAC=lactic acid; LAS=acid soluble lignin; M=mannose; n/a=not applicable; nd=not detected; nr=not requested; SUC=succinic acid; TS=total solids; X=xylose; XYL=xylitol

Name(s) of CAT Staff Working on Project:	Reviewed by:
Ray Ruiz	

CHEMICAL ANALYSIS & TESTING

Analytical Report

Results and Comments

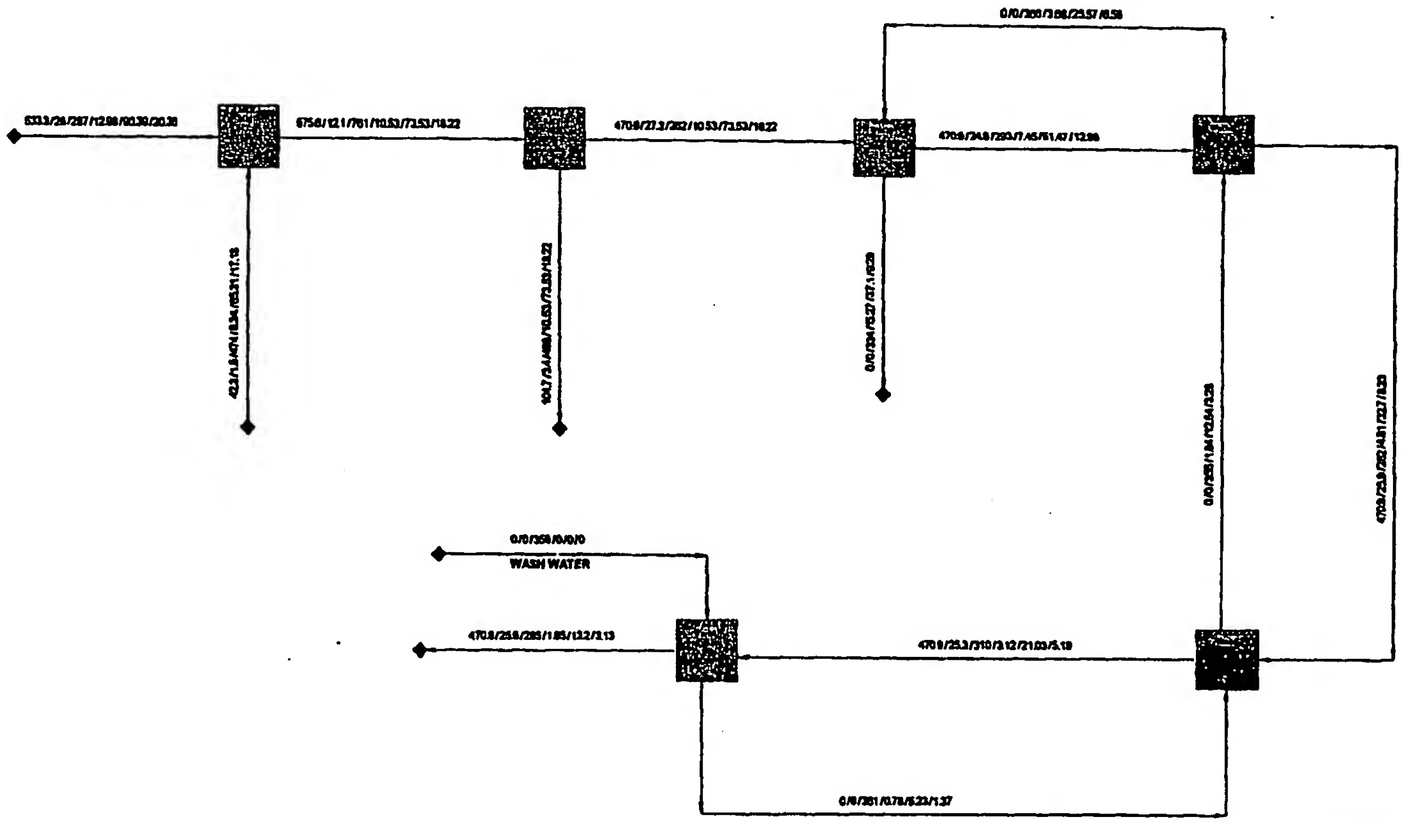
	Sample	[X] mg/ml C	X	AA	[X] % Dry Weight TS
25	Test #6 Run #3 2nd Stage Filtrate	5.03	35.18	8.98	5.34
26	Test #6 Run #3 3rd Stage Filtrate	3.17	22.08	5.76	3.45
27	Test #6 Run #3 4th Stage Filtrate	1.50	10.26	2.81	1.67
28	Test #6 Run #1 1st Stage Filtrate	5.68	40.19	10.20	6.12
29	Test #6 Run #1 2nd Stage Filtrate	4.48	31.64	8.20	4.78
30	Test #6 Run #1 3rd Stage Filtrate	2.43	16.97	4.48	2.63
31	Test #6 Run #1 4th Stage Filtrate	1.10	7.54	2.06	1.26
32	Test #6 Run #2 1st Stage Filtrate	5.87	41.58	10.47	6.35
33	Test #6 Run #2 2nd Stage Filtrate	3.79	26.71	6.95	4.13
34	Test #6 Run #2 3rd Stage Filtrate	2.28	15.94	4.20	2.55
35	Test #6 Run #2 4th Stage Filtrate	1.05	7.17	1.93	1.23
36	Test #6 Run #3 1st Stage Filtrate	5.34	37.83	9.62	5.77
37	Test #6 Run #3 2nd Stage Filtrate	3.85	27.12	7.00	4.29
38	Test #6 Run #3 3rd Stage Filtrate	2.20	15.31	4.03	2.45
39	Test #6 Run #3 4th Stage Filtrate	0.96	6.53	1.82	1.18
40	Test #7 Run #1 1st Stage Filtrate	5.94	41.87	10.55	6.44
41	Test #7 Run #1 2nd Stage Filtrate	3.71	26.07	6.69	4.04
42	Test #7 Run #1 3rd Stage Filtrate	1.79	12.33	3.23	2.05
43	Test #7 Run #1 4th Stage Filtrate	0.85	5.62	1.39	1.06
44	Test #7 Run #2 1st Stage Filtrate	5.50	38.70	9.88	6.00
45	Test #7 Run #2 2nd Stage Filtrate	3.77	26.37	6.76	4.13
46	Test #7 Run #2 3rd Stage Filtrate	1.78	12.23	3.22	2.03
47	Test #7 Run #2 4th Stage Filtrate	0.65	4.41	1.16	0.85
48	Test #7 Run #3 1st Stage Filtrate	4.69	33.16	7.99	5.14
49	Test #7 Run #3 2nd Stage Filtrate	3.52	24.63	6.32	3.92
50	Test #7 Run #3 3rd Stage Filtrate	1.91	13.19	3.42	2.22
51	Test #7 Run #3 4th Stage Filtrate	0.86	5.76	1.56	1.02
52	Test #8 Run #1 1st Stage Filtrate	6.16	43.48	11.16	6.71
53	Test #8 Run #1 2nd Stage Filtrate	5.13	36.34	9.20	5.58
54	Test #8 Run #1 3rd Stage Filtrate	3.14	22.08	5.81	3.47
55	Test #8 Run #1 4th Stage Filtrate	1.71	11.81	3.17	2.00
56	Test #8 Run #2 1st Stage Filtrate	5.70	40.28	10.12	6.37
57	Test #8 Run #2 2nd Stage Filtrate	4.89	34.58	8.82	5.40
58	Test #8 Run #2 3rd Stage Filtrate	2.94	20.59	5.36	3.33
59	Test #8 Run #2 4th Stage Filtrate	1.51	10.43	2.80	1.79
60	Test #8 Run #3 1st Stage Filtrate	4.94	34.97	8.85	5.39
61	Test #8 Run #3 2nd Stage Filtrate	5.19	36.77	9.37	5.76
62	Test #8 Run #3 3rd Stage Filtrate	3.08	21.58	5.54	3.47
63	Test #8 Run #3 4th Stage Filtrate	1.59	11.08	3.02	1.89

A=arabinose; AA=acetic acid; CEL=cellobiose; ET=ethanol; FA=formic acid; FL=furfural; G=glucose; GA=galactose; GLY=glycerol; HMF=5-hydroxymethyl-2-furaldehyde; LA=levulinic acid; LAC=lactic acid; LAS=acid soluble lignin; M=mannose; n/a=not applicable; nd=not detected; nr=not requested; SUC=succinic acid; TS=total solids; X=xylose; XYL=xylitol

HARRIS GROUP INC.

SEATTLE, WA

LABORATORY TRIAL



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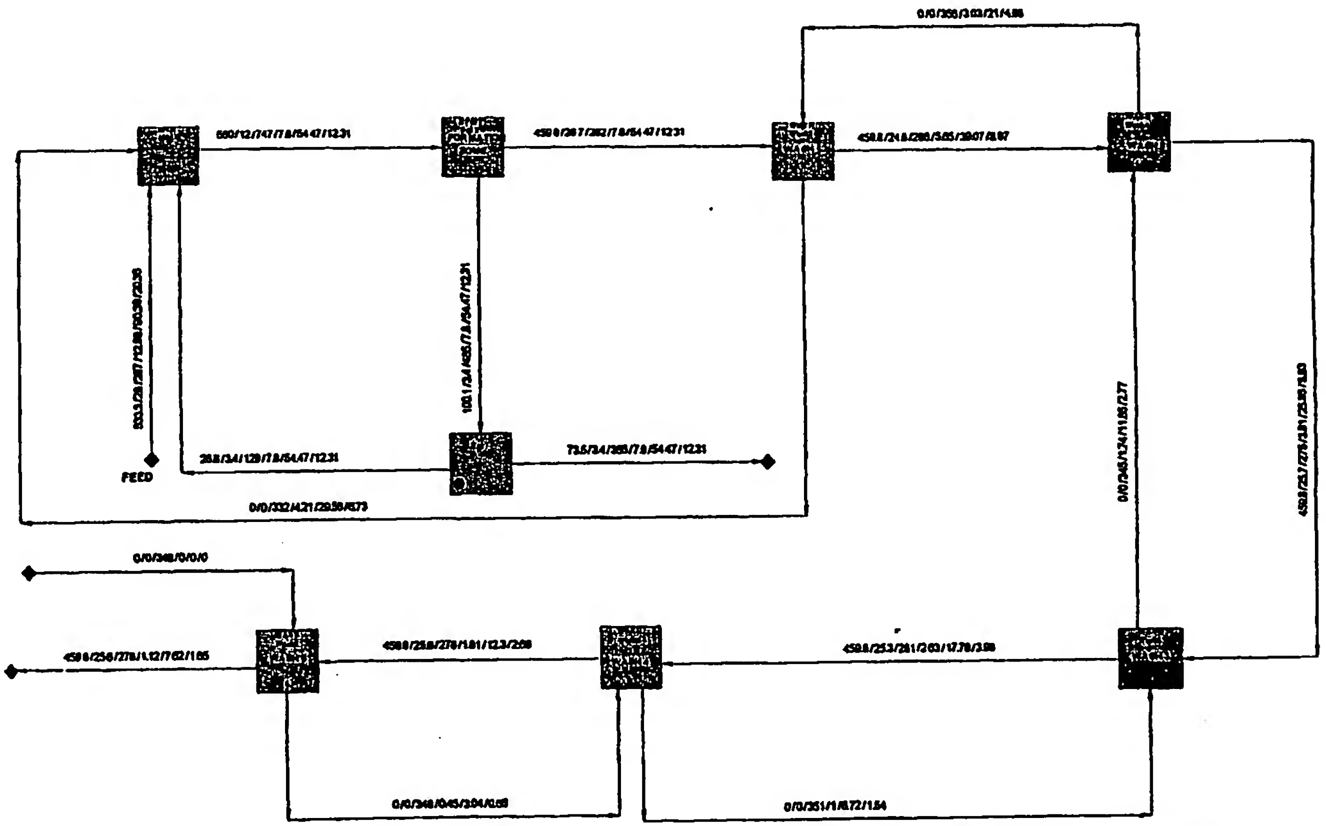
HARRIS GROUP INC.

SEATTLE, WA

PROCESS 100 533.3 BDST/DAY



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HARRIS GROUP INC.

SEATTLE, WA

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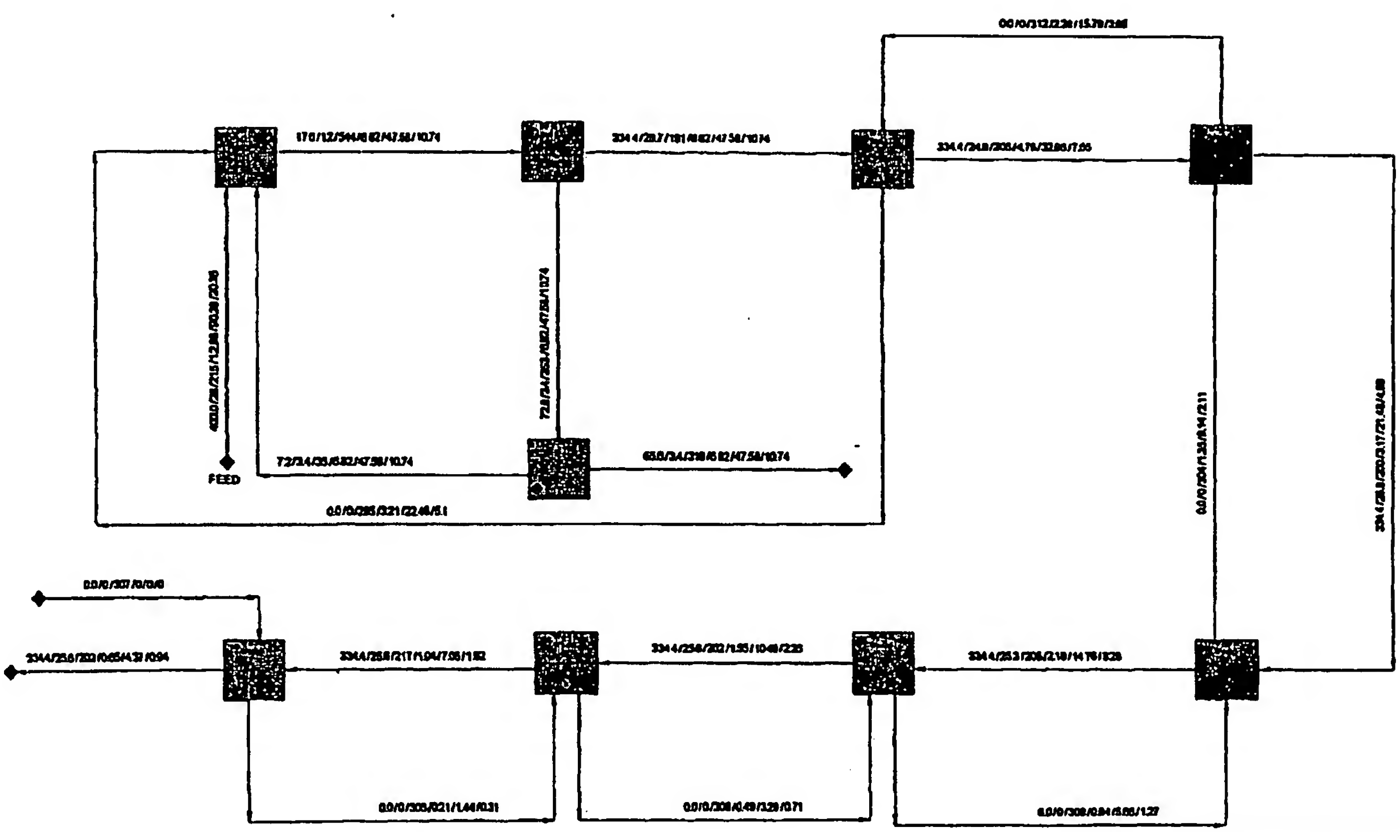

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CHEMICAL ANALYSIS & TESTING

Analytical Report

Report No.	Page
2000-032A	1 of 2

Project Title: BlackClawson Liquids

NREL In-House	Current Subcontractor	CRADA	Other
[X]	[ ]	[ ]	[ ]

Name of Project Contact Person	Mark Ruth	Date Sample Delivered	01/27/2000
NREL Notebook	NB# 2275, pp 048-051	Date Work Completed	02/04/2000
Sample Number and Type	63 liquor samples	Actual Hours Spent	12
Summary of Work Required	Analysis of liquids for total solids content and for free soluble glucose, xylose, and acetic-acid.	Proposed Analysis	Analyzed in-house according to NREL Laboratory Analytical Procedures.

Work required: Sample Prep [ ] Acid Digest [ ] HPLC [X] YSI [ ] GC [ ] CHN [ ] Other: [ ]

Results and Comments

Sample	[X] mg/ml G	X	AA	[X] % Dry Weight TS
1 Forming Zone Filtrate	10.95	76.44	18.09	nr
2 Dilution Liquor	9.34	65.31	17.18	nr
3 First Stage Shower	3.77	26.28	6.82	nr
4 Second Stage Shower	1.62	11.06	2.93	nr
5 Third Stage Shower	0.76	4.98	1.34	nr
6 Fresh Water (4th Stage Shower)	nd	nd	nd	nr
7 Test 02 Run 01 1st Stage Filtrate	4.75	32.82	8.01	5.23
8 Test 02 Run 02 1st Stage Filtrate	5.36	38.00	9.67	5.86
9 Test 02 Run 03 1st Stage Filtrate	5.79	40.66	10.50	6.22
10 Test 03 Run 01 1st Stage Filtrate	4.19	28.79	7.16	4.47
11 Test 03 Run 02 1st Stage Filtrate	4.50	31.88	8.06	4.99
12 Test 03 Run 03 1st Stage Filtrate	4.93	35.06	8.99	5.37
13 Test 04 Run 01 1st Stage Filtrate	5.09	36.12	8.92	5.56
14 Test 04 Run 02 1st Stage Filtrate	5.25	37.35	9.64	5.73
15 Test 04 Run 03 1st Stage Filtrate	3.99	28.23	7.42	4.41
16 Test 05 Run 01 1st Stage Filtrate	6.17	42.32	10.52	6.58
17 Test 05 Run 01 2nd Stage Filtrate	5.75	39.63	9.86	6.03
18 Test 05 Run 01 3rd Stage Filtrate	3.32	22.92	5.86	3.58
19 Test 05 Run 01 4th Stage Filtrate	1.50	10.09	2.74	1.70
20 Test 05 Run 02 1st Stage Filtrate	5.89	41.38	10.53	6.37
21 Test 05 Run 02 2nd Stage Filtrate	5.19	36.63	9.35	5.59
22 Test 05 Run 02 3rd Stage Filtrate	2.97	20.89	5.33	3.29
23 Test 05 Run 02 4th Stage Filtrate	1.48	10.22	2.82	1.78
24 Test 05 Run 03 1st Stage Filtrate	5.28	36.80	9.17	5.71

A=arabinose; AA=acetic acid; CEL=cellobiose; ET=ethanol; FA=formic acid; FL=furfural; G=glucose; GA=galactose; GLY=glycerol; HMF=5-hydroxymethyl-2-furaldehyde; LA=levulinic acid; LAC=lactic acid; LAS=acid soluble lignin; M=mannose; n/a=not applicable; nd=not detected; nr=not requested; SUC=succinic acid; TS=total solids; X=xylose; XYL=xylitol

Name(s) of CAT Staff Working on Project:	Reviewed by:
Ray Ruiz	

CHEMICAL ANALYSIS & TESTING

Analytical Report

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Results and Comments

	Sample	☒ mg/ml G	X	AA		☒ % Dry Weight TS	
25	Test Ø5 Run Ø3 2nd Stage Filtrate	5.03	35.18	8.98		5.34	
26	Test Ø5 Run Ø3 3rd Stage Filtrate	3.17	22.08	5.76		3.45	
27	Test Ø5 Run Ø3 4th Stage Filtrate	1.50	10.26	2.81		1.67	
28	Test Ø6 Run Ø1 1st Stage Filtrate	5.68	40.19	10.20		6.12	5
29	Test Ø6 Run Ø1 2nd Stage Filtrate	4.48	31.64	8.20		4.78	
30	Test Ø6 Run Ø1 3rd Stage Filtrate	2.43	16.97	4.48		2.63	
31	Test Ø6 Run Ø1 4th Stage Filtrate	1.10	7.54	2.06		1.26	
32	Test Ø6 Run Ø2 1st Stage Filtrate	5.87	41.58	10.47		6.35	6
33	Test Ø6 Run Ø2 2nd Stage Filtrate	3.79	26.71	6.95		4.13	
34	Test Ø6 Run Ø2 3rd Stage Filtrate	2.28	15.94	4.20		2.55	
35	Test Ø6 Run Ø2 4th Stage Filtrate	1.05	7.17	1.93		1.23	
36	Test Ø6 Run Ø3 1st Stage Filtrate	5.34	37.83	9.62		5.77	6
37	Test Ø6 Run Ø3 2nd Stage Filtrate	3.85	27.12	7.00		4.29	
38	Test Ø6 Run Ø3 3rd Stage Filtrate	2.20	15.31	4.03		2.45	
39	Test Ø6 Run Ø3 4th Stage Filtrate	0.96	6.53	1.82		1.18	
40	Test Ø7 Run Ø1 1st Stage Filtrate	5.94	41.87	10.55		6.44	
41	Test Ø7 Run Ø1 2nd Stage Filtrate	3.71	26.07	6.69		4.04	
42	Test Ø7 Run Ø1 3rd Stage Filtrate	1.79	12.33	3.23		2.05	
43	Test Ø7 Run Ø1 4th Stage Filtrate	0.85	5.62	1.39		1.06	
44	Test Ø7 Run Ø2 1st Stage Filtrate	5.50	38.70	9.88		6.00	
45	Test Ø7 Run Ø2 2nd Stage Filtrate	3.77	26.37	6.76		4.13	
46	Test Ø7 Run Ø2 3rd Stage Filtrate	1.78	12.23	3.22		2.03	
47	Test Ø7 Run Ø2 4th Stage Filtrate	0.65	4.41	1.16		0.85	
48	Test Ø7 Run Ø3 1st Stage Filtrate	4.69	33.16	7.99		5.14	
49	Test Ø7 Run Ø3 2nd Stage Filtrate	3.52	24.63	6.32		3.92	
50	Test Ø7 Run Ø3 3rd Stage Filtrate	1.91	13.19	3.42		2.22	
51	Test Ø7 Run Ø3 4th Stage Filtrate	0.86	5.76	1.56		1.02	
52	Test Ø8 Run Ø1 1st Stage Filtrate	6.16	43.48	11.16		6.71	
53	Test Ø8 Run Ø1 2nd Stage Filtrate	5.13	36.34	9.20		5.58	
54	Test Ø8 Run Ø1 3rd Stage Filtrate	3.14	22.08	5.81		3.47	
55	Test Ø8 Run Ø1 4th Stage Filtrate	1.71	11.81	3.17		2.00	
56	Test Ø8 Run Ø2 1st Stage Filtrate	5.70	40.28	10.12		6.37	
57	Test Ø8 Run Ø2 2nd Stage Filtrate	4.89	34.58	8.82		5.40	
58	Test Ø8 Run Ø2 3rd Stage Filtrate	2.94	20.59	5.36		3.33	
59	Test Ø8 Run Ø2 4th Stage Filtrate	1.51	10.43	2.80		1.79	
60	Test Ø8 Run Ø3 1st Stage Filtrate	4.94	34.97	8.85		5.39	
61	Test Ø8 Run Ø3 2nd Stage Filtrate	5.19	36.77	9.37		5.76	
62	Test Ø8 Run Ø3 3rd Stage Filtrate	3.08	21.58	5.54		3.47	
63	Test Ø8 Run Ø3 4th Stage Filtrate	1.59	11.08	3.02		1.89	

A=arabinose; AA=acetic acid; CEL=cellobiose; ET=ethanol; FA=formic acid; FL=furfural; G=glucose; GA=galactose; GLY=glycerol; HMF=5-hydroxymethyl-2-furaldehyde; LA=levulinic acid; LAC=lactic acid; LAS=acid soluble lignin; M=mannose; n/a=not applicable; nd=not detected; nr=not requested; SUC=succinic acid; TS=total solids; X=xylose; XYL=xylitol

CHEMICAL ANALYSIS & TESTING

Analytical Report

[illegible]	Page
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BlackClawson Solids

NREL In-House [X] Current Subcontractor CRADA Other [ ]

[illegible]	Mark Ruth	[illegible]	01/27/2000
[illegible]	NB# 2275, pp 048-051	[illegible]	02/04/2000
[illegible]	21 washed solids and 1 "biomass as received" solid	[illegible]	20
[illegible]	Analysis of solids for total solids content and for entrained glucose, xylose, and acetic-acid.	[illegible]	Analyzed in-house according to NREL Laboratory Analytical Procedures.

Work required: Sample Prep [ ] Acid Digest [ ] HPLC [X] YSI [ ] GC [ ] CHN [ ] Other: [ ]

Results and Comments

	[X] mg/ml				[X] % Dry Weight
Sample	G	X	AA		TS
1 Washed Solids 1 stage Test #2 Run #1	3.49	23.55	5.73		31.58
2 Washed Solids 1 stage Test #2 Run #2	3.40	23.50	6.19		32.00
3 Washed Solids 1 stage Test #2 Run #3	3.79	26.31	6.92		31.58
4 Washed Solids 1 stage Test #3 Run #1	4.10	27.67	7.10		38.76
5 Washed Solids 1 stage Test #3 Run #2	2.42	16.42	4.31		29.84
6 Washed Solids 1 stage Test #3 Run #3	2.27	15.40	3.98		29.75
7 Washed Solids 1 stage Test #4 Run #1	2.40	16.35	4.02		30.42
8 Washed Solids 1 stage Test #4 Run #2	2.18	14.75	3.71		30.73
9 Washed Solids 1 stage Test #4 Run #3	1.88	12.50	3.13		30.77
10 Washed Pulp 4 stage Test #6 Run #1	0.76	4.75	1.21		28.11
11 Washed Solids 4 stage Test #6 Run #2	1.08	7.18	1.80		29.38
12 Washed Pulp 4 stage Test #6 Run #3	0.94	6.05	1.50		27.39
13 Washed Solids 4 stage CC washing Test #6 Run #1	0.64	3.89	0.98		27.84
14 Washed Solids 4 stage CC washing Test #6 Run #2	1.06	6.90	1.61		29.47
15 Washed Solids Test #6 Run #3	1.05	6.87	1.60		28.27
16 Washed Solids 4 stage countercurrent washing Test #7 Run #1	0.64	3.77	0.87		28.18
17 Washed Solids 4 stage washing 1.1 lb H2O / lb solids as received Test #7 Run #2	0.77	4.71	1.07		29.11
18 Washed Solids Test #7 Run #3	0.86	5.45	1.24		28.39
19 Washed Solids 4 stage Test #8 Run #1	1.54	10.54	2.52		29.39
20 Washed Solids 4 stage Test #8 Run #2	1.17	7.68	1.91		29.02
21 Washed Solids 4 stage Test #8 Run #3	1.30	0.71	2.06		28.39
22 Biomass as received	12.98	90.39	20.36		38.13

A=arabinose; AA=acetic acid; CEL=cellobiose; ET=ethanol; FA=formic acid; FL=furfural; G=glucose; GA=galactose; GLY=glycerol; HMF=5-hydroxymethyl-2-furaldehyde; LA=levulinic acid; LAC=lactic acid; LAS=acid soluble lignin; M=mannose; n/a=not applicable; nd=not detected; nr=not requested; SUC=succinic acid; TS=total solids; X=xylose; XYL=xylitol

Name(s) of CAT Staff Working on Project: Ray Ruiz

Reviewed by:

APPENDIX D
PNEUMAPRESS

HARRIS GROUP INC.
PROJECT NO: 99-10600
REVISION A 07-Jun-01

NATIONAL RENEWABLE ENERGY LABORATORY
GOLDEN, COLORADO
PNEUMAPRESS POST DISTILLATE LIQUID SOLID SEPARATION

MECHANICAL EQUIPMENT LIST

EQUIP # REV MOTOR #	DESCRIPTION	VENDOR P.O. ISSUED	SIZE CAPACITY HEAD	GEAR RATIO	EQUIP STATUS	HORSEPOWER RPM VOLTS	ENCLOSURE FRAME MODEL NO.	REMARKS
F-101	PNEUMAPRESS No. 1	Pneumapress Filter Corp	MOD 30-10-316 285 GPM 300 SQ FT AREA			60 HP		316 SS WETTED COMPONENTS
F-102	PNEUMAPRESS No. 2	Pneumapress Filter Corp.	MOD 30-10-316 285 GPM 300 SQ FT AREA			60 HP		316 SS WETTED COMPONENTS
F-103	PNEUMAPRESS No. 3	Pneumapress Filter Corp.	MOD 30-10-316 285 GPM 300 SQ FT AREA			60 HP		316 SS WETTED COMPONENTS
F-104	PNEUMAPRESS No. 4	Pneumapress Filter Corp.	MOD 30-11-316 314 GPM 314 SQ FT AREA			60 HP		316 SS WETTED COMPONENTS
CV-101	DISCHARGE CONVEYOR		4' WIDE X 60' LONG			50 HP		
P-101	PNEUMAPRESS FEED PUMP No. 1	GOULDS	MOD 3410 10X12-17 4800 GPM 200 FT TDH			300 HP 1780 RPM		IRON CASING W/316 SS TRIM
P-102	PNEUMAPRESS FEED PUMP No. 2	GOULDS	4800 GPM 200 FT TDH			300 HP 1780 RPM		IRON CASING W/316 SS TRIM
T-101	FILTRATE TANK		12' -6" DIA X 13' FT H 12,000 GAL					316 SS CONSTRUCTION
P-103	FILTRATE PUMP	GOULDS	MOD 3196 6X8-13 1300 GPM 70 FT TDH					316 SS CONSTRUCTION
C-101	AIR COMPRESSOR - CENTRIFUGAL #1	Cooper	1435 ACFM 125 PSIG			300 HP		
C-102	AIR COMPRESSOR - CENTRIFUGAL #2	Cooper	1435 ACFM 125 PSIG			300 HP		
T-103	AIR RECEIVER #1		1000 GAL 150 PSIG					
T-104	AIR RECEIVER #2		1000 GAL 150 PSIG					
T-105	AIR RECEIVER #3		1000 GAL 150 PSIG					
T-106	AIR RECEIVER #4		1000 GAL 150 PSIG					
					Total	1490 HP		

HARRIS GROUP INC.
PROJECT NO: 99-10600
REVISION B 04-Feb-01

NATIONAL RENEWABLE ENERGY LABORATORY
GOLDEN, COLORADO
PNEUMAPRESS - PROCESS 100 - LIQUID SOLID SEPARATION

MECHANICAL EQUIPMENT LIST

EQUIP # REV MOTOR #	DESCRIPTION	VENDOR P.O. ISSUED	SIZE CAPACITY HEAD	GEAR RATIO	EQUIP STATUS	HORSEPOWER RPM VOLTS	ENCLOSURE FRAME MODEL NO.	REMARKS
F-101	PNEUMAPRESS No. 1	Pneumapress Filter Corp	MOD 30-12-316 285 GPM 360 SQ FT AREA			60 HP		316 SS WETTED COMPONENTS
F-102	PNEUMAPRESS No. 2	Pneumapress Filter Corp.	MOD 30-12-316 285 GPM 360 SQ FT AREA			60 HP		316 SS WETTED COMPONENTS
F-103	PNEUMAPRESS No. 3	Pneumapress Filter Corp.	MOD 30-12-316 285 GPM 360 SQ FT AREA			60 HP		316 SS WETTED COMPONENTS
CV-101	DISCHARGE CONVEYOR		4' WIDE X 60' LONG			50 HP		
P-101	PNEUMAPRESS FEED PUMP No. 1	GOULDS	MOD 3410 10X12-17 4800 GPM 200 FT TDH			300 HP 1780 RPM		316 SS CONSTRUCTION
P-102 B	PNEUMAPRESS FEED PUMP No. 2	GOULDS	MOD 3410 10X12-17 4800 GPM 200 FT TDH			300 HP 1780 RPM		316 SS CONSTRUCTION
T-101 B	PRIMARY FILTRATE TANK		10" DIA X 11' FT H 6000 GAL					316 SS CONSTRUCTION
P-103	PRIMARY FILTRATE PUMP	GOULDS	MOD 3410 6 x 8 -14 1000 GPM 60 FT TDH			25 HP		316 SS CONSTRUCTION
T-106 B	WASH FILTRATE TANK		10" DIA X 11' FT H 6000 GAL					316 SS CONSTRUCTION
P-104 B	WASH FILTRATE PUMP	GOULDS	MOD 3410 10X12-17 6000 GPM 200 FT TDH			400 HP		316 SS CONSTRUCTION
T-107 B	PRESS FEED TANK		10" DIA X 11' FT H 6000 GAL					316 SS CONSTRUCTION
A-101	PRESS FEED TANK AGITATOR					25 HP		RUBBER LINED
C-101	AIR COMPRESSOR - CENTRIFUGAL #1	COOPER	1180 ACFM 125 PSIG			250 HP		CENTRIFUGAL TYPE AIR COMPRESSOR
C-102	AIR COMPRESSOR - CENTRIFUGAL #2	COOPER	1180 ACFM 125 PSIG			250 HP		CENTRIFUGAL TYPE AIR COMPRESSOR
T-103	AIR RECEIVER #1		1000 GAL 150 PSIG					
T-104	AIR RECEIVER #2		1000 GAL 150 PSIG					
T-105	AIR RECEIVER #3		1000 GAL 150 PSIG					

HARRIS GROUP INC.
PROJECT NO: 99-10600
REVISION B 04-Feb-01

NATIONAL RENEWABLE ENERGY LABORATORY
GOLDEN, COLORADO
PNEUMAPRESS - PROCESS 300 - LIQUID SOLID SEPARATION

MECHANICAL EQUIPMENT LIST

EQUIP # REV MOTOR #	DESCRIPTION	VENDOR P.O. ISSUED	SIZE CAPACITY HEAD	GEAR RATIO	EQUIP STATUS	HORSEPOWER RPM VOLTS	ENCLOSURE FRAME MODEL NO.	REMARKS
F-101	PNEUMAPRESS No. 1	Pneumapress Filter Corp	MOD 30-12-316 285 GPM 360 SQ FT AREA			60 HP		316 SS WETTED COMPONENTS
F-102	PNEUMAPRESS No. 2	Pneumapress Filter Corp.	MOD 30-12-316 285 GPM 360 SQ FT AREA			60 HP		316 SS WETTED COMPONENTS
F-103	PNEUMAPRESS No. 3	Pneumapress Filter Corp.	MOD 30-12-316 285 GPM 360 SQ FT AREA			60 HP		316 SS WETTED COMPONENTS
CV-101	DISCHARGE CONVEYOR		4' WIDE X 60' LONG			50 HP		
P-101	PNEUMAPRESS FEED PUMP No. 1	GOULDS	MOD 3410 10X12-17 4800 GPM 200 FT TDH			300 HP 1780 RPM		316 SS CONSTRUCTION
P-102 B	PNEUMAPRESS FEED PUMP No. 2	GOULDS	MOD 3410 10X12-17 4800 GPM 200 FT TDH			300 HP 1780 RPM		316 SS CONSTRUCTION
T-101 B	PRIMARY FILTRATE TANK		10" DIA X 11' FT H 6000 GAL					316 SS CONSTRUCTION
P-103	PRIMARY FILTRATE PUMP	GOULDS	MOD 3410 6 x 8 -14 1000 GPM 60 FT TDH			25 HP		316 SS CONSTRUCTION
T-106 B	WASH FILTRATE TANK		10" DIA X 11' FT H 6000 GAL					316 SS CONSTRUCTION
P-104 B	WASH FILTRATE PUMP	GOULDS	MOD 3410 10X12-17 6000 GPM 200 FT TDH			400 HP		316 SS CONSTRUCTION
T-107 B	PRESS FEED TANK		10" DIA X 11' FT H 6000 GAL					316 SS CONSTRUCTION
A-101	PRESS FEED TANK AGITATOR					25 HP		RUBBER LINED
C-101	AIR COMPRESSOR - CENTRIFUGAL #1	COOPER	1180 ACFM 125 PSIG			250 HP		CENTRIFUGAL TYPE AIR COMPRESSOR
C-102	AIR COMPRESSOR - CENTRIFUGAL #2	COOPER	1180 ACFM 125 PSIG			250 HP		CENTRIFUGAL TYPE AIR COMPRESSOR
T-103	AIR RECEIVER #1		1000 GAL 150 PSIG					
T-104	AIR RECEIVER #2		1000 GAL 150 PSIG					
T-105	AIR RECEIVER #3		1000 GAL 150 PSIG					

going to be sent upstream the Chemi-Washer, it will be necessary to include a fines-liquid separator (such as a Dissolved Air Flotation clarifier) to treat such stream and prevent excessive fines buildup around the washing loop. Devices (e.g. belt press) for thickening the separated fines should be also included.

It was also found that multiple Chemi-Washers® will be needed to achieve the production target.

Based on the data generated on the sample supplied and applying our sizing criteria, we recommend the following:

- For Process 100, three (3) Chemi-Washers®, 8 meters wide by 20 meters long, with 5 washing stages, will be able to perform the acetic acid removal at the desired efficiency. The fresh water addition will be ~1.1 lb / lb FSAR.
- For Process 300, four (4) Chemi-Washers®, 10 meters wide by 22 meters long, with 6 washing stages will perform a 95% sugars removal. The fresh water addition should be approx. 1.4 lb water / lb FSAR.

Because of the FSAR's acidic nature and its temperature (100°C), the construction material for the liquor- and biomass-exposed parts for either Process should be 317L stainless steel.

Guillermo Dietrich-V.
Sr. Research Engineer

Michael A. Sieron
Vice-President, Sales

David E. Chupka
Manager of Technology

Report Number: 41,031 Date: April 6, 2000

Counter-Current Washing Trials on Bagasse Biomass

OBJECTIVE

The objective of this trial was to simulate the operation of a commercial Chemi-Washer® by using a lab-scale drainage tester and determine the washing efficiency of a one and a four-stage countercurrent washing operation.

BACKGROUND

The Chemi-Washer® is a counter-current, dissolved solids washer which can afford high removal efficiency of dissolved solids in a fiber slurry with minimum fresh water requirements.

The operational advantages of the Chemi-Washer® are:

(a) Low dilution factor, typically 1.0.
(b) High spent liquor concentration.
(c) High washing efficiency.
(d) Excellent turn down ratio at sustained high efficiency.
(e) Small space requirements.
(f) No intermediate filtrate tanks.
(g) Simple fan for vacuum generation.
(h) A wire life under normal operating conditions of 7 months.

During the Chemi-Washer operation, the pulp is pumped into a Headbox for distribution onto a pin seam fabric wire. The fabric transports the pulp from the Headbox to the discharge end of the machine. Under the wire, from the Headbox to the discharge end of the machine, suction boxes collect all the liquid which is displaced through the pulp. The area between the Headbox and the first shower, called the formation zone, is designed to dewater the pulp from inlet consistency to displacement consistency..

Once the pulp mat is formed, it passes under a set of showers where the filtrate from the succeeding washing stage gently flows onto the pulp mat. By controlling the flow to the last shower, the dilution factor is set to the desired value -approximately 1.0-, to ensure a high level of dissolved solids in the spent liquor. The pulp is not re-diluted and mixed between washing stages, so displacement washing is the mechanism for recovery of the dissolved solids in the spent liquor. Displacement washing permits the fulfillment of two major washing objectives: low dilution factor and high washing efficiency.

Stock dewatering and washing is accomplished by an integrated suction box-shower pump-centrifugal fan-hood arrangement. The suction boxes of each washing stage are connected to the shower pumps. A level controller maintains a vapor phase within

each suction box and controls the flow to the shower of the preceding stage. The vapor from each suction box is drawn into a centrifugal fan and recycled back into the hood. A pressure differential between the hood and the vapor phase of the suction box, provided by the fan, forces the liquor to pass from the stock to the suction box. This pressure differential is normally in the range of 0.6 to 1.0 meters of water.

The hood maintains a seal between the atmosphere and the suction boxes so the machine is virtually odor-free. The hood also prevents cooling of the pulp and shower liquor by the ambient air. This higher temperature improves the drainage characteristics of the pulp.

Harris Group Inc. charged Thermo Black Clawson Inc. to determine the size and operating conditions of one or several Chemi-Washers able to handle a production of 1600 oven-dry tons per day of bagasse biomass at 27% consistency. The final, washed pulp should meet the following specifications:

- For one process (Process 100), the amount of acetic acid present in the washed pulp's liquor at 27% consistency should be a maximum of 3.3 g/Kg liquor; the preferable limit is 1.65 g/Kg liquor.
- For another process (Process 300), the sugars (glucose and xylan) removal efficiency - based on concentration in the incoming FSAR-, should be 95% minimum; the preferable limit is 98%. This figure applies to a washed pulp consistency of 27%.

EXPERIMENTAL PROCEDURE

The drainage testing trials were performed in the week of January 12 to January 19, 2000. Mr. John Lukas from the Harris Group was present during the first two trial days.

Sample and dilution liquor preparation :

A 55-gallon drum of FSAR was sent for testing. The sample has a texture similar to paper mill's sludge. A consistency determination was performed on a kneaded sample from both top and middle of the drum. According to the process flowsheets, the consistency should be between 26 and 27 percent, which was similar to the sample in the middle. Since this will be the feed to the Chemi-Washer, water was added to make a visually pumpable suspension.

Because of the process' dilution water limitations, the only way we could make a pumpable suspension was to mix some of the filtrate from the forming zone back with the FSAR.
A simulated steady-state dilution liquor was prepared by adding 1.5 parts of water to one part of FSAR, followed by mixing, settling and filtration under vacuum using a standard Chemi-Washer (Duratech 2000) wire. The filtrate collected during this operation was used to dilute a second, fresh sample of FSAR and the process repeated two more times. After the third concentrating stage, the liquor's pH was measured and found to be within 0.2 units of the FSAR's liquid phase.
The final liquor, having a suspended solids content of 1.45 percent, was identified as the "dilution liquor".

Drainage Testing Procedure :

A lab-scale, drainage tester was used for these trials. A sketch of this apparatus is shown in Figure 1.

The feed pulp was prepared by weighing a 67.2 grams of FSAR and then adding, with mixing, 100.8 grams of dilution liquor. This pulp had a theoretical suspended solids content of 12.1%, including the solids from the dilution liquor (11.2% excluding solids from dilution liquor). Table I shows the testing matrix requested by the customer.

Table I : Drainage Testing Matrix

Test Number	Process	Washing Stages	Fresh Water Usage	Fresh Water Temperature
1	---	No Wash	---	---
2	100	1	0.59 lb / lb FSAR	117°F
3	300	1	0.86 lb / lb FSAR	145°F
4	300	1	1.11 lb / lb FSAR	145°F
5	100	4	0.59 lb / lb FSAR	117°F
6	300	4	0.86 lb / lb FSAR	145°F
7	300	4	1.11 lb / lb FSAR	145°F
8	300	4	0.59 lb / lb FSAR	145°F

Formation Zone :

To simulate drainage in the formation zone, the feed pulp was heated to 212°F (100°C) and poured onto the wire in the drainage tester which was filled with warm water

Counter-Current Washing Trials on Bagasse Biomass

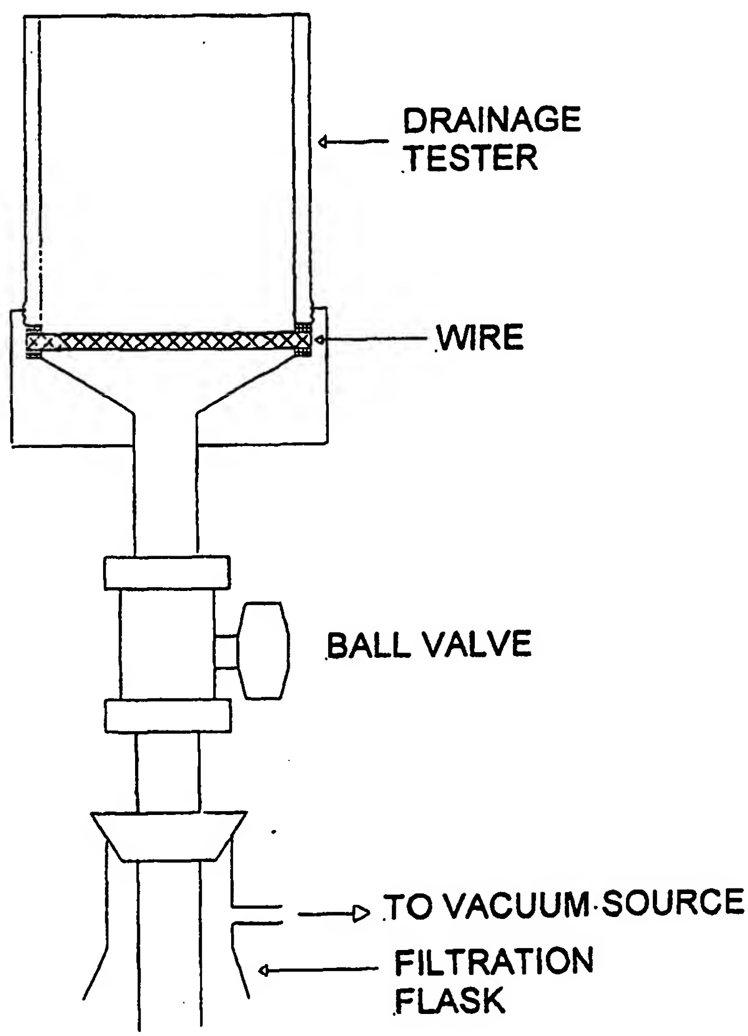

FIG. 1 : SKETCH OF DRAINAGE TESTER

to just cover the wire. The vacuum level in the filtration flask was set to 7 inches of water. The ball valve was then opened and the time required to get a "dry line" was recorded using a stopwatch.

Washing Stages:

After performing the forming zone filtration, the vacuum was shut down, the drainage tester detached from the filtration flask and the diluted formation zone filtrate was discarded.

The one or four-stage washing procedure was performed as follows:

(a) Re-attach the drainage tester to the filtration flask.
(b) Set the vacuum level.
(c) Place a given amount of shower liquor/fresh water (40 grams for runs 2, 5 and 8; 58 grams for runs 3 and 6; 75 grams for runs 4 and 7) at a fixed temperature on top of the pulp mat with minimal disruption.
(d) Open the ball valve.
(e) Using a stopwatch, record the time required to get a dry line.
(f) Close the ball valve and shut the vacuum.
(g) Detach the drainage tester from the filtration flask.
(h) Collect the filtrate from this stage in a plastic bottle. Seal the bottle, label it and set it aside for shipping.
(i) If more washing stages should be performed, then repeat stages (a) through (h).

Triplicate runs for each one of the tests were performed. For tests 5 through 8, a simulated shower liquor for the 1st, 2nd and 3rd stages was prepared according to our proprietary testing protocol. Tap water at the stated temperature was used as shower for the 4th stage. The shower liquor temperature was set with a 14°F (8°C) gradient between the fresh water and the first stage shower. Obviously, no temperature gradient was established for runs 2 through 4.

For a given test, the final pulp pad at the end of each run was collected, weighed and split into two approximately equal parts. One part was again weighed and a consistency test was run on it. The other part was placed in a plastic bag, sealed, labeled and set aside for shipment. From the initial weight and consistency of the washed pulp, the discharge suspended solids inventory was determined.

Preparation of constant streams and formation zone filtrate :

Throughout all the tests, the composition of the shower liquors, dilution liquor and fresh water was kept constant. Samples of these streams (including filtered dilution liquor) were poured into plastic bottles, labeled and set aside for shipment.

To prepare the formation zone filtrate, five Chemi-Washer feed samples were prepared according with the procedure described above. Each sample was then heated to 212 °F and drained according to the "Forming Zone" procedure with the exception that no water was loaded to the drainage tester. An aliquot (430 grams) of the combined, 5 run filtrate was split into three parts. Each part was then filtered through a tared filter paper under vacuum until the filtrate was clear. This required two "passes". The suspended solids collected in the filter paper were dried to constant weight at 107°C. A suspended solids balance was then performed. A sample of the combined clear filtrates was poured into a plastic bottle, labeled as "formation zone filtrate" and set aside for shipment.

Analytical testing :

The following analysis were preformed in the Technology Center's laboratory:

1. Specific Gravity of all filtrates, showers and dilution liquor.
2. pH of all filtrates, showers, dilution liquor and FSAR's liquid phase.
3. Consistencies of the dilution liquor, washed pulp and formation zone filtrate.
4. Bauer McNett (TAPPI T 233 method) and 0.006" slot Pulmac debris analysis on the FSAR.

All individual filtrates and washed pulps, constant streams, filtered filtration zone filtrate and FSAR were sent to the NREL laboratory in Golden, CO for analysis of dry solids, glucose, xylan and acetic acid content.

RESULTS AND DISCUSSION

FSAR :

The consistency of the top and middle portions in the 55-gallon drum of FSAR were 32.2% and 28.1% respectively. According to the process data, the consistency of FSAR should be between 26 and 27 percent. Therefore the middle portion of the sample was used for the drainage test trials.

The Bauer McNett analysis on the FSAR yielded the following results:

Table II: Bauer McNett analysis on FSAR

Solids retained on 28 mesh screen	24.9 %
Solids retained on 60 mesh screen	8.5 %
Solids retained on 100 mesh screen	11.8 %
Solids retained on 200 mesh screen	10.5 %
Fines	44.3 %

On visual inspection, all the material retained on 28 mesh screen consisted mainly of chop pieces and shives. The amount of debris (chop, shives etc.) was quantified with a Pulmac Shives Analyzer equipped with a 0.006" (0.15 mm) slot screen plate. The average debris was 26.7% based on oven dry-solids. Table III shows the calculated composition of the FSAR's fiber fraction excluding the debris. Such fiber length distribution is similar to a paper mill's sludge, which normally show very poor drainage characteristics.

Table III: Bauer McNett analysis of FSAR's fiber fraction alone

Solids retained on 28 mesh screen	0.0
Solids retained on 60 mesh screen	9.2
Solids retained on 100 mesh screen	16.1
Solids retained on 200 mesh screen	14.3
Fines	60.4

Drainage Rates :

The Data Sheets containing information of temperatures, vacuum levels, drainage times, amount of filtrate collected and discharge consistency for all the tests are found in the Appendix. The area available for drainage in the tester is 74 cm^2.

As expected, the FSAR showed very poor drainage characteristics. The calculated drainage rates were less than 1.6 gallons per minute per square feet of drainage area in some washing zones.

Specific Gravity and pH :

Tables IV to VII list the pH and Specific Gravity results. To minimize loss of volatile compounds, the specific gravity was measured wit liquors at room temperature.

Counter-Current Washing Trials on Bagasse Biomass

Table IV : pH and Specific Gravity
Filtrates from 1st Stage at Room Temperature

TEST #	RUN #	pH	Specific Gravity
2	1	1.8	1.00
	2	1.7	1.01
	3	1.7	1.01
3	1	1.9	1.00
	2	1.8	1.00
	3	1.8	1.01
4	1	1.8	1.01
	2	1.9	1.01
	3	1.8	1.01

Table V: pH and Specific Gravity of Constant Streams
Tests 2 through 8

Stream	Temp. °F	pH	Sp. Gr.
Fresh Water	145	7.7	0.98
3rd Stage Shower	122	2.7	0.99
	149	2.5	0.98
2nd Stage Shower	127	2.3	0.99
	153	2.1	0.98
1st Stage Shower	133	2.2	1.00
	158	2.0	1.00
FZ Filtrate	176	1.6	1.02
Dilution Liquor	176	1.7	1.02

Counter-Current Washing Trials on Bagasse Biomass

Table VI: pH of Filtrates at Room Temperature

TEST #	RUN #	1st Stage	2nd Stage	3rd Stage	4th Stage
5	1	1.9	1.9	2.1	2.4
	2	1.9	1.9	2.1	2.3
	3	1.9	2.0	2.1	2.3
6	1	2.2	2.3	2.5	2.8
	2	2.2	2.3	2.5	2.7
	3	2.2	2.3	2.5	2.8
7	1	2.4	2.6	2.8	3.0
	2	2.5	2.6	2.8	3.0
	3	2.5	2.6	2.8	3.0
8	1	2.0	2.0	2.1	2.3
	2	2.0	2.0	2.1	2.3
	3	2.0	2.0	2.1	2.3

Table VII: Specific Gravity of Filtrates at Room Temperature

TEST #	RUN #	1st Stage	2nd Stage	3rd Stage	4th Stage
5	1	1.01	1.01	1.00	0.99
	2	1.02	1.01	1.00	0.99
	3	1.02	1.01	1.00	0.99
6	1	1.01	1.00	1.00	0.99
	2	1.01	1.00	0.99	0.99
	3	1.01	1.00	1.00	1.00
7	1	1.02	1.00	0.99	0.99
	2	1.02	1.01	1.00	0.99
	3	1.01	1.01	1.00	0.99
8	1	1.01	1.01	1.00	0.99
	2	1.01	1.01	1.00	0.99
	3	1.01	1.02	1.00	0.99

Sugars and Acetic Acid analyses :

A detailed report of sugars (glucose and xylan) and acetic acid concentration in all samples -performed at the NREL laboratory-, can be found in the Appendix. A summary of those results is given below. The following abbreviations are used:

G = Glucose
X = Xylan
AA = Acetic Acid.

Liquors:

Table VIII shows the average composition of the different Liquors for all the trials. The composition of the constant streams is included for comparison purposes (filtrate of the *(n)th* stage becomes the shower of the *(n-1)th* stage).

For tests 5 through 8, there is a very good agreement on the sugars and acetic acid concentrations in filtrates and showers when using 0.86 and 1.11 lb water/lb FSAR. However for the tests using 0.59 lb water/lb FSAR, the concentration in the showers are approximately half of the corresponding for filtrates.
This anomaly can be explained by using the concept of Dilution Factor. The Dilution Factor is defined as the weight of water entering the liquor system per unit weight of oven-dry solids washed. For practical purposes, we can assume that the water leaving with the washed pulp came entirely from the last shower and it can be assessed by using the water-to-fiber ratio (W/F), defined as

$$W/F = \frac{100 - \%C}{\%C}$$

Therefore the dilution factor (DF) can be calculated as follows:

$$DF = \frac{Water - (Fiber)(W/F)}{Fiber}$$

Where "Fiber" and "Water" are the weights of oven-dry fiber and water used during washing.

Assuming a discharge consistency of 27%, the W/F will be 73/27 = 2.704 and the calculated dilution factors for the different amounts of wash water are:

THERMO BLACK CLAWSON INC.

A Thermo Fibertek company

Report Number: 41,031

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Table VIII: Summary of Liquor Analyses

STREAM	Filtrates Average mg/l			Showers Average mg/l		
	G	X	AA	G	X	AA
Forming Zone Filtrate	10.95	76.44	18.09			
Dilution Liquor	9.34	65.31	17.18			
Test #2, 1st Stage Filtrate	5.30	37.16	9.39	0.00	0.00	0.00
Test #3, 1st Stage Filtrate	4.54	31.91	8.07	0.00	0.00	0.00
Test #4, 1st Stage Filtrate	4.78	33.90	8.66	0.00	0.00	0.00
TEST # 5						
1st Stage	5.78	40.20	10.07	3.77	26.28	6.82
2nd Stage	5.32	37.15	9.40	1.62	11.06	2.93
3rd Stage	3.15	21.97	5.65	0.76	4.98	1.34
4th Stage	1.49	10.19	2.79	0.00	0.00	0.00
TEST # 6						
1st Stage	5.63	39.87	10.09	3.77	26.28	6.82
2nd Stage	4.04	28.49	7.38	1.62	11.06	2.93
3rd Stage	2.30	16.07	4.24	0.76	4.98	1.34
4th Stage	1.04	7.08	1.94	0.00	0.00	0.00
TEST # 7						
1st Stage	5.38	37.91	9.47	3.77	26.28	6.82
2nd Stage	3.67	25.69	6.59	1.62	11.06	2.93
3rd Stage	1.83	12.59	3.29	0.76	4.98	1.34
4th Stage	0.78	5.26	1.37	0.00	0.00	0.00
TEST # 8						
1st Stage	5.60	39.57	10.04	3.77	26.28	6.82
2nd Stage	5.07	35.90	9.13	1.62	11.06	2.93
3rd Stage	3.05	21.41	5.57	0.76	4.98	1.34
4th Stage	1.61	11.11	3.00	0.00	0.00	0.00

$$DF_{0.59} = \frac{0.59 - 0.27(2.704)}{0.27} \approx -0.52$$

$$DF_{0.86} = \frac{0.86 - 0.27(2.704)}{0.27} \approx 0.48$$

$$DF_{1.11} = \frac{1.11 - 0.27(2.704)}{0.27} \approx 1.41$$

With 0.59 lb water/lb FSAR, the flow rate of shower liquor in the *(n)th* stage is not enough to displace the pulp's liquor from the *(n-1)th* stage. Under this condition, there always will be liquor carryover from the *(n-1)th* stage going to the *(n+1)th* stage and the washing operation is hindered. The amount of fresh water required to achieve a DF of zero (that is, to just theoretically displace the liquor from the previous stage) is 0.73 lb / lb of FSAR.

The Chemi-Washer normally operates with a Dilution Factor of 1.0, which will require the usage of 1.0 lb water/lb FSAR for effective washing

Pulp Pads and Washing Efficiency :

Table IX shows a summary of the pulp pads analyses and the calculated washing efficiency results based on the data provided by NREL. Their consistency data were significantly higher than ours. Perhaps this difference was due to the fact that some of the pulp pad's liquor had to be squeezed out of the sample for analysis prior to running the consistency test. We felt that our consistency data for such samples was more reliable.

Based on the customer specifications and the analysis of acetic acid and sugars in the FSAR, the removal efficiencies of such entities should be:

- For process 100, the minimum removal efficiency is 83.8% [(20.36 – 3.3)/20.36]; preferably 91.9% [(20.36 – 1.65)/20.36] at a discharge consistency of 27%.
- For Process 300, the minimum sugar removal efficiency is 95% or 0.65 g glucose and 4.52 g xylan per Kilogram in the final pulp's liquor at 27% consistency.

From our lab-generated data and the liquor's analyses, we ran a series of WINGEMS simulations at different fresh water additions on a four-stage countercurrent washer. In general, we found an excellent agreement between the reported data and the

WINGEMS-generated data. The only exception was again found in the sugars and acetic acid concentrations in the final pulp pads. According to WINGEMS, the concentration of such entities should be 2.4 to 2.7 times higher than that reported.

From the data on Table IX, we can see that the customer's specifications for the final pulp could be reached with a 4-stage washer operating at a DF of 1.4 (1.11 lb water / lb FSAR). However, as mentioned before, WINGEMS rendered a different set of data for the washed pulp. Table X shows the WINGEMS-predicted concentrations of sugars and acetic acid in the washed pulp's liquor under our lab conditions as a function of dilution factor

Table X: Concentrations predicted by WINGEMS in washed pulp. 4-Stage countercurrent washing at different Dilution Factors

Fresh Water lb / lb FSAR	Dilution Factor	G mg/ml	X mg/ml	AA mg/ml
0.73	0.0	2.64	18.05	4.34
0.87	0.5	2.33	15.87	3.79
1.00	1.0	2.12	14.39	3.42
1.14	1.5	1.97	13.34	3.16

From table X we can notice that a 4-stage washer will render a washing efficiency marginal for acetic acid but unacceptable for sugars. The simulation data at 1.14 lb of fresh water per pound FSAR is shown in the Appendix.

In "real mode" operation, the dilution liquor must be provided by pumping all the filtrate from the first stage plus some of the formation zone filtrate back to the Chemi-Washer's Headbox. Since a 4-stage washer is just marginal for acetic acid removal and we already have enough fresh water addition, we should try a 5-stage washer. Table XI shows the WINGEMS-predicted concentrations of sugars and acetic acid in the final pulp's liquor using a 5-stage washer as a function of the fresh water addition.

Table IX: Washing Efficiency Calculations based on NREL data

	Average, mg/ml In Pulp's Liquor			%	Average, g / Kg OD solids			Washing Eficiency, %			
	G	X	AA	C	G	X	AA	G	X	AA	Ave.
Biomass as Received	12.98	90.39	20.36	28.0	33.38	232.43	52.36				
Washed Solids, Test # 2	3.56	24.45	6.28	25.8	10.23	70.32	18.07	69.3	69.7	65.5	68.2
Washed Solids, Test # 3	2.93	19.83	5.13	26.2	8.25	55.86	14.44	75.3	76.0	72.4	74.6
Washed Solids, Test # 4	2.15	14.53	3.62	26.8	5.88	39.69	9.89	82.4	82.9	81.1	82.1
Washed Solids, Test # 5	0.93	5.99	1.50	25.7	2.68	17.33	4.35	92.0	92.5	91.7	92.1
Washed Solids, Test # 6	0.92	5.89	1.39	26.1	2.59	16.67	3.94	92.2	92.8	92.5	92.5
Washed Solids, Test # 7	0.76	4.64	1.06	26.8	2.07	12.68	2.90	93.8	94.5	94.5	94.3
Washed Solids, Test # 8	1.34	8.98	2.17	26.2	3.77	25.28	6.10	88.7	89.1	88.4	88.7

Table XI: Concentrations predicted by WINGEMS in washed pulp. "Real mode" operation with 5-Stage countercurrent washing

Fresh Water lb / lb FSAR	Dilution Factor	G mg/ml	X mg/ml	AA mg/ml
0.73	0.0	2.3	15.9	3.5
0.87	0.5	1.8	12.1	2.6
1.00	1.0	1.4	9.6	2.1
1.14	1.5	1.2	7.9	1.7

From table XI we can see that the specifications for Process 100 are met even at a dilution factor of zero. Therefore a 5-stage Chemi-Washer will be perform satisfactorily. A WINGEMS simulation under these conditions is also attached in the Appendix.

According to Table XI, the washing efficiency for sugars with a 5-stage washer is still low, around 91 percent, at a dilution factor of 1.5. To meet the required efficiency for sugars we have to add another washing stage.
Using WINGEMS to simulate a 6-stage washer, we found that the specifications for sugars can be met by using 1.41 lb of fresh water per pound of FSAR, equivalent to a dilution factor of 2.5. This additional washing stage, coupled with the increased filtrate handling requirements (24 percent more as compared to a dilution factor of 1.5) translates into a much larger drainage table area. A WINGEMS simulation with these conditions is also attached in the Appendix.

Solids loss in Formation Zone :

From the Data Sheets, the grand average weight of the washed pulp pads was 15.6 oven-dry grams. For each drainage test, we used 18.9 oven-dry grams (67.2*0.281) of pulp. Therefore 3.3 grams of solids (mostly fines) were loss in the Formation Zone, equivalent to 17.5% of the initial load.

The suspended solids content (by triplicate) of the Forming Zone filtrate was 6.08%. A mass balance around this zone, knowing that the 100.8*0.0145 = 1.46 grams of suspended solids are being loaded by the dilution liquor, yielded a solids loss of 19%.

The solids carried in the Formation Zone filtrate are composed mostly by fines, which are very difficult to retain by either the pulp mat or the wire.

If the excess liquor from the Formation Zone is sent upstream the Chemi-Washer, then a device should be installed (for example, a *Dissolved Air Flotation clarifier*) to separate the fines from such liquor and avoid a "doom loop" of fines around the washing process.

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CHEMI-WASHER SIZING

According with the process data provided, we should design a Chemi-Washer able to wash, to the required efficiency, 478,700 pounds per hour of biomass at 27.8% solids or ~1,600 oven-dry short tons per day.

Based on the drainage rate data and using our proprietary sizing criteria, the production requirements will not be achieved with one single machine. Since the washing efficiency specifications are different for acetic acid and sugars, the best options are:

1. For Process 100, use three (3) Chemi-Washers, 8 meters wide by 20 meters long running with 5 washing stages at a dilution factors 1.0 to 1.5.
2. For Process 300, use four (4) Chemi-Washers, 10 meters wide by 22 meters long running with 6 washing stages at a dilution factor of 2.5

For either Process, the dilution water (1.5 lb water / lb FSAR) required to pump the FSAR to the Chemi-Washer's Headbox will be supplied by mixing all the filtrate from the 1st stage and some of the Forming Zone filtrate to the FSAR. The excess Forming Zone filtrate with its associated suspended solids should be fed to a fines-liquid separator, such as a Dissolved Air Flotation clarifier to prevent fines buildup in the washing loop. A sludge thickener should also be installed to extract as much as possible of such liquor in the separated sludge.

Because of the acidic properties and temperature of the FSAR, all of the Chemi-Washer's wetted parts as well as the hood should be build of 317L stainless steel.

THERMO BLACK CLAWSON INC.

A Thermo Fibertek company

Report Number: 41,031

Date: April 6, 2000

Counter-Current Washing Trials on Bagasse Biomass

APPENDIX

CHEMIWASHER DRAINAGE TEST DATA SHEET

Company	Harris Group Inc.	%C as received	28.1	Total Solids in liquor, %	
Location	Seattle, WA	DS in pulp's liquor		Suspended Solids in Dil. Liq., %	1.45
Pulp Type	Fermented Bagasse	Pulp's CSF, ml		Sp. Grav. Dilution Liqour:	1.02 @ 80 ° C
Kappa Number		Additional Sample Data	Test # 2 . 0.59 lb Fresh Water @ 48°C / lb FSAR		

		FEED SLURRY			FORMING ZONE		1ST STAGE		2ND STAGE		3RD STAGE	
Run No.	Date	Weight, grams	Consist. %	Temp. °C	Temp. °C	Drain Time, Secs.	Temp. °C	Drain Time, Secs.	Temp. °C	Drain Time, Secs.	Temp. °C	Drain Time, Secs.
1	1/17/00	168	11.2	100	97	6.5	48	5.1	--	--	--	--
2	1/17/00	168	11.2	98	96	6.3	48	4.7	--	--	--	--
3	1/17/00	168	11.2	100	97	6.2	48	5.3	--	--	--	--
AVERAGE →						6.3	48	5.0				

4TH STAGE		5TH STAGE	
Temp. °C	Drain Time, Secs.	Temp. °C	Drain Time, Secs.
--	--	--	--
--	--	--	--
--	--	--	--

REMARKS:

Filtrate Collected: 53.4 g, 42.9 g, 4937 g. Average = 48.7 g

Discharge Solids: 25.6%, 25.2%, 26.7%. Average = 25.8%

←AVERAGE

ZONE VACUUMS (inches of water)

F.Z.	8	4th Stage	
1st Stage	30		
2nd Stage			
3rd Stage			

WASH VOLUME, ml

1st Stage	40
2nd Stage	
3rd Stage	
4th Stage	

Tested by	GDV
Location	TBC Tech. Ctr.

Project # 41,031

CHEMIWASHER DRAINAGE TEST DATA SHEET

Company	Harris Group Inc.	%C as received	28.1	Total Solids in liquor, %	
Location	Seattle, WA	DS in pulp's liquor		Suspended Solids in Dil. Liq., %	1.45
Pulp Type	Fermented Bagasse	Pulp's CSF, ml		Sp. Grav. Dilution Liqour:	1.02 @ 80 ° C
Kappa Number		Additional Sample Data	Test # 3. 0.86 lb. Fresh Water @ 64 °C / lb FSAR		

		FEED SLURRY			FORMING ZONE		1ST STAGE		2ND STAGE		3RD STAGE	
Run No.	Date	Weight, grams	Consist. %	Temp. °C	Temp. °C	Drain Time, Secs.	Temp. °C	Drain Time, Secs.	Temp. °C	Drain Time, Secs.	Temp. °C	Drain Time, Secs.
1	1/17/00	168	11.2	99	96	7.0	63	5.2	--	--	--	--
2	1/17/00	168	11.2	100	96	7.1	63	5.5	--	--	--	--
3	1/17/00	168	11.2	98	96	6.2	63	6.0	--	--	--	--
AVERAGE →						6.8		5.6				

4th STAGE		5TH STAGE	
Temp. °C	Drain Time, Secs.	Temp. °C	Drain Time, Secs.
--	--	--	--
--	--	--	--
--	--	--	--

REMARKS:

Filtrate Collected: 66.0g, 63.8 g, 67.3 g. Average = 65.7 g

Discharge Consistency: 25.9%, 27.0%, 25.7%. Average = 26.2%

←AVERAGE

ZONE VACUUMS (inches of water)		
F.Z.	10	4th Stage
1st Stage	30	
2nd Stage		
3rd Stage		

WASH VOLUME, ml	
1st Stage	58
2nd Stage	58
3rd Stage	
4th Stage	

Tested by	GDV
Location	TBC Tech. Ctr.

Project # 41,031

PNEUMAPRESS® FILTER CORPORATION

301 Ohio Avenue
Richmond, CA 94804
Phone (510) 232-2653
Fax (510) 232-5228

May 30, 2000

John Lukas
Harris Group Inc.
1000 Denny Way, Suite 800
Seattle, WA 98109-5338

RE: Wood Chip Hydrolyzate Filter Equipment (Ref. HGI/PFC E-mail 05/17/00 regarding NREL Tests)

Dear John,

Today I am out of the office and do not have the NREL Test Plans for process information or the test results mentioned in your e-mail. I will ask our office to provide these to me before I leave for the Midwest.

To make sure we are talking about the same tests, I have with me test #1, #2, #3, #4, (#5 was changed to #7), and #7A from 01/27/00 and 01/28/00. I also have Test #8 and Test #9 from 02/09/00, and Post Distillate Tests D1 and D2 from 01/28/00.

Since I do not have sizing criteria with me today, I can at least give and example of throughput using Test #9:

Cycle Description:

Slurry Fill ______________________ 5-15 seconds
Liquid Clear #1 = 3 seconds (assume 5 seconds.... 5 seconds to include equipment actuation).
Liquid Clear #2 = 2 seconds (use 5 seconds) ...... 5 seconds.
Liquid Clear #3 = 2.5 seconds (use 5 seconds) .....5 seconds.
Blowdown ______________________ 30 seconds.
Plates Open/Close and Cake Discharge ....20 – 30 seconds.
Total Cycle is 70 – 90 seconds *

*Use 75 – 100 seconds to be conservative.

144/3.14 x 100 ml mother slurry is processed per square foot every 1 minute 15 seconds (minimum) to 1 minute 40 seconds (maximum). This comes out to 43.6 gallons/ft^2/hr to 58.2 gallon/ft^2/hr mother slurry being processed depending on NREL process requirements.

I wanted to provide the above information up front to make sure we are thinking the same. I believe NREL will be at the Corn Utilization & Technology Conference in Minneapolis at the same time I will. Looking forward to talking to you this week.

Best regards,

Steve C. Benesi

Steve C. Benesi

JAN-16-1900 15:00

---PNEUMAPRESS® FILTER CORPORATION

500 gm solids
750 ml. Hydrolyzate filtrate

BENCH TEST DATA SHEET
FOR
PNEUMAPRESS® TEST CYCLINDER

DATE: 1/27/00 TIME: ______ TEST RUN # 1

	DATA		REMARKS
COMPANY	Harris Group		251.4 85°C Slurry
SAMPLE I.D.	Wood Chip Hydrolyzate		
SLURRY QUANTITY	100 ml		
SLURRY % OF SOLIDS	~10%		
BLOW DOWN PRESSURE	100 psi		
TIME FOR FILTRATE TO CLEAR	< 5 sec.		
BLOW DOWN TIME	30 sec.		
AMOUNT OF FILTRATE	55 ml filtrate		Clear
CAKE THICKNESS	3/4"		
TARE + CAKE WEIGHT (FRESH)			
	1/HR (SOLID)	2/HR (BROKEN)	
TARE + CAKE WEIGHT (DRY)			
TARE WEIGHT			
	1/HR (SOLID)	2/HR (BROKEN)	
CAKE WEIGHT (DRY)			
MOISTURE % IN FRESH CAKE			

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BENCH TEST DATA SHEET
FOR
PNEUMAPRESS® TEST CYCLINDER

DATE: 1/27/00 TIME: ______ TEST RUN # 2

	DATA		REMARKS
COMPANY	HARRIS GROUP		
SAMPLE I.D.	Wood Chip Hydrolyzate		
SLURRY QUANTITY	100 ml		85°C / tap H_2O @ 47°C 20 ml
SLURRY % OF SOLIDS	~10%		
BLOW DOWN PRESSURE	100 psi		
TIME FOR FILTRATE TO CLEAR	3.5 sec. clear, then		1 sec wash to go to clr.
BLOW DOWN TIME	30 seconds		
AMOUNT OF FILTRATE	44 ml mother filtrate		/ 27.5 ml wash filtrate
CAKE THICKNESS	7/8"		
TARE + CAKE WEIGHT (FRESH)	38 gr. – fresh cake only –		
TARE + CAKE WEIGHT (DRY)	1/HR (SOLID)	2/HR (BROKEN)	
TARE WEIGHT			
CAKE WEIGHT (DRY)	1/HR (SOLID)	2/HR (BROKEN)	
MOISTURE % IN FRESH CAKE			

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BENCH TEST DATA SHEET
FOR
PNEUMAPRESS® TEST CYLINDER

DATE: 1/27/00 TIME: ______ TEST RUN # 3

	DATA		REMARKS
COMPANY	Harris Group		
SAMPLE I.D.	Wood Chips Hydrolyzate		
SLURRY QUANTITY	100 ml		85°C / 30 ml wash 125°C
SLURRY % OF SOLIDS	~10%		
BLOW DOWN PRESSURE	100 psi		
TIME FOR FILTRATE TO CLEAR	3.5 seconds mother filtrate		4 seconds wash filtrate
BLOW DOWN TIME	30 second		
AMOUNT OF FILTRATE	mother filtrate - 42 ml		Wash filtrate: 44 ml
CAKE THICKNESS	1/8"		→ filter media not tightened all the way - RETEST -
TARE + CAKE WEIGHT (FRESH)	36.5 gr. cake.		
TARE + CAKE WEIGHT (DRY)	1/HR (SOLID)	2/HR (BROKEN)	
TARE WEIGHT			
CAKE WEIGHT (DRY)	1/HR (SOLID)	2/HR (BROKEN)	
MOISTURE % IN FRESH CAKE			

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BENCH TEST DATA SHEET FOR PNEUMAPRESS® TEST CYCLINDER

DATE: 1/27/00 TIME: ______ TEST RUN # 4

	DATA		REMARKS
COMPANY	Harris Group		@ 5K media
SAMPLE I.D.	Wood Chips Hydrolyzate		all tests
SLURRY QUANTITY	100 ml 85°C		37 ml wash @ 145°F
SLURRY % OF SOLIDS	~10%		
BLOW DOWN PRESSURE	100 psi		
TIME FOR FILTRATE TO CLEAR	~~Mother~~ 4 secs.		5 sec.
BLOW DOWN TIME	30 sec.		
AMOUNT OF FILTRATE	32 ml mother		52 ml wash
CAKE THICKNESS	15/16"		
TARE + CAKE WEIGHT (FRESH)	39g - Fresh cake		
TARE + CAKE WEIGHT (DRY)	1/HR (SOLID)	2/HR (BROKEN)	
TARE WEIGHT			
CAKE WEIGHT (DRY)	1/HR (SOLID)	2/HR (BROKEN)	
MOISTURE % IN FRESH CAKE			

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BENCH TEST DATA SHEET FOR PNEUMAPRESS® TEST CYCLINDER

DATE: 1/28/00 TIME: ______ TEST RUN # 5 7

	DATA				REMARKS
COMPANY	Harris Group				GD5K media
SAMPLE I.D.	Wood Chip Hydrolyzate				78-80° C
	Two-Stage -				
SLURRY QUANTITY	100 ml				Wash #1 ≤ 43.22cc H_2O/1.17cc filtrate - Wash #2 ≤
SLURRY % OF SOLIDS	~10%				
BLOW DOWN PRESSURE	100 psi				
TIME FOR FILTRATE TO CLEAR	mother 4 secs	1st wash 5 secs.	2nd wash 5 sec	3rd wash 5 sec ±	
BLOW DOWN TIME	30 seconds				
AMOUNT OF FILTRATE	mother 38 ml	1st wash 57 ml	2nd wash 44 ml	3rd wash 50 ml	
CAKE THICKNESS	3/4"				
TARE + CAKE WEIGHT (FRESH)	* Cake only = 26 1/2 g.				* cake lumpified probably from sticky chunks
TARE + CAKE WEIGHT (DRY)	1/HR (SOLID)	2/HR (BROKEN)			
TARE WEIGHT					
CAKE WEIGHT (DRY)	1/HR (SOLID)	2/HR (BROKEN)			
MOISTURE % IN FRESH CAKE					

NOTE: washes @ ~50-55° C

PNEUMAPRESS® FILTER CORPORATION

BENCH TEST DATA SHEET
FOR
PNEUMAPRESS® TEST CYCLINDER

DATE: 1/28/00 TIME: ______ TEST RUN # 7A

	DATA		REMARKS
COMPANY	Harris Group		0.5K-media
SAMPLE I.D.	Wood Chip Hydrolysate		
SLURRY QUANTITY	100 ml —		wash #1 43 ml H_2O/1 ml Filtrate; wash #2 - 44 ml H_2O -
SLURRY % OF SOLIDS	~10%		
BLOW DOWN PRESSURE	100 psi		
TIME FOR FILTRATE TO CLEAR	mother 3.5 / wash #1 / wash #2		
BLOW DOWN TIME	[illegible] ml / [illegible] ml / [illegible] ml		
AMOUNT OF FILTRATE	54 ml		
CAKE THICKNESS	~ 3/4 - 7/8"		
TARE + CAKE WEIGHT (FRESH)	fresh cake 27.8 g.		Dry pan ck. — fresh cake + 4 g. tare = 9.1 g.
TARE + CAKE WEIGHT (DRY)	1/HR (SOLID)	2/HR (BROKEN)	Final 6.3 g - $\frac{6.3g - 4}{9.1g - 4}$
TARE WEIGHT			45.1% solids
CAKE WEIGHT (DRY)	1/HR (SOLID)	2/HR (BROKEN)	54.9% moist.
MOISTURE % IN FRESH CAKE			

JAN-16-1900 15:02

PNEUMAPRESS® FILTER CORPORATION

BENCH TEST DATA SHEET FOR PNEUMAPRESS® TEST CYCLINDER

DATE: 2/9/00 TIME: ________ TEST RUN # 8

	DATA			REMARKS
COMPANY	HARRIS GROUP			GUSK
SAMPLE I.D.	WOOD CHIP HYDROLYZATE			~80-85°C
SLURRY QUANTITY	100 ml		wash #1 34.65 H_2O .94 filtrate 35.59 ml	wash #2 35.59 H_2O
SLURRY % OF SOLIDS	~10%			
BLOW DOWN PRESSURE	100 psi			
TIME FOR FILTRATE TO CLEAR	mother 3-4 sec	wash #1 3 sec	wash #2 3 sec	
BLOW DOWN TIME	—	—	30 sec	
AMOUNT OF FILTRATE	mother 45 mls	wash #1 38 mls	wash #2 43 mls	CLEAR
CAKE THICKNESS	3/4" – appears "washed" & relatively dry.			
TARE + CAKE WEIGHT (FRESH)	CAKE ONLY = 22 g.			
TARE + CAKE WEIGHT (DRY)	1/HR (SOLID)	2/HR (BROKEN)		
TARE WEIGHT				
CAKE WEIGHT (DRY)	1/HR (SOLID)	2/HR (BROKEN)		
MOISTURE % IN FRESH CAKE				

...FILTER CORPORATION

BENCH TEST DATA SHEET
FOR
PNEUMAPRESS® TEST CYCLINDER

DATE: 2/9/00 TIME: ________ TEST RUN # 9

	DATA			REMARKS
COMPANY	Harris Group			GDSTC
SAMPLE I.D.	Wood Chip Hydrolyzate			~80-85°C
SLURRY QUANTITY	100 ml	Wash #1 22.97 H_2O 0.62 filtrate 23.59		Wash #2 23.59 H_2O
SLURRY % OF SOLIDS	~10%			
BLOW DOWN PRESSURE	100 psi			
TIME FOR FILTRATE TO CLEAR	mother 3 sec	Wash #1 2 sec	Wash #2 2-3 sec	
BLOW DOWN TIME	—	—	30 sec.	
AMOUNT OF FILTRATE	mother 48 ml	Wash #1 25 ml	Wash #2 35 ml	
CAKE THICKNESS	5/8+"			
TARE + CAKE WEIGHT (FRESH)	21 g. Cake only			
TARE + CAKE WEIGHT (DRY)	1/HR (SOLID)	2/HR (BROKEN)		
TARE WEIGHT				
CAKE WEIGHT (DRY)	1/HR (SOLID)	2/HR (BROKEN)		
MOISTURE % IN FRESH CAKE				

PNEUMAPRESS® FILTER CORPORATION

BENCH TEST DATA SHEET
FOR
PNEUMAPRESS® TEST CYCLINDER

DATE: 1/28/00

TIME: ______

TEST RUN # D 1

	DATA		REMARKS
COMPANY	Harris Group		GDSK
SAMPLE I.D.	POST Distillate		80-85°C
SLURRY QUANTITY	240 ml ±		
SLURRY % OF SOLIDS			
BLOW DOWN PRESSURE	100 psi		
TIME FOR FILTRATE TO CLEAR	2 min.		
BLOW DOWN TIME	1 min		
AMOUNT OF FILTRATE	205 ml filtrate		– clear (dark amber)
CAKE THICKNESS	1/2" ~		
TARE + CAKE WEIGHT (FRESH)	22.1 g.		
TARE + CAKE WEIGHT (DRY)	1/HR (SOLID)	2/HR (BROKEN)	
TARE WEIGHT			
CAKE WEIGHT (DRY)	1/HR (SOLID)	2/HR (BROKEN)	
MOISTURE % IN FRESH CAKE			

JAN-16-1900 15:03

PNEUMAPRESS® FILTER CORPORATION

BENCH TEST DATA SHEET
FOR
PNEUMAPRESS® TEST CYCLINDER

DATE: 1/28/00 TIME: ______ TEST RUN # D2

	DATA		REMARKS
COMPANY	Harris Group		GDSK
SAMPLE I.D.	Post DISTILLATE		
SLURRY QUANTITY	100 ml		
SLURRY % OF SOLIDS			
BLOW DOWN PRESSURE	100 psi		
TIME FOR FILTRATE TO CLEAR	20 sec.		
BLOW DOWN TIME	30 sec.		
AMOUNT OF FILTRATE	85 ml.		
CAKE THICKNESS	5/32"		
TARE + CAKE WEIGHT (FRESH)	Cake only 8g.		
TARE + CAKE WEIGHT (DRY)	1/HR (SOLID)	2/HR (BROKEN)	
TARE WEIGHT			
CAKE WEIGHT (DRY)	1/HR (SOLID)	2/HR (BROKEN)	
MOISTURE % IN FRESH CAKE			

Rah'u'a
99-10600/11
2/00

		.86	
wash	1.11	0.88	0.69
3			
water	44.4	35.6	23.6
filt	0		
tot	44.4	35.6	23.6
2			
water	44.16	0.188	23.47
filt	0.23	35.41	0.125
tot	44.39	35.598	23.595
1			
water	43.22	34.65	22.97
filt	1.17	0.94	0.62
tot	44.39	35.59	23.59

WASH 1

WASH 2 = H_2O

water

85°C

3-4 sec 45 mls
3 sec. 38 mls
3 sec. 43 mls
22g. cake
appears fairly dry.
3/4"

GDSK.

3 - 48 ml 2 washers
2 - 25 ml
2-3 - 35 ml not 3 washes
21.2 g. cake
5/8"
Label Well

CHEMICAL ANALYSIS & TESTING

Analytical Report

Analysis No.	Page
2000-041 S&L	1 of 3

Project Title	Pneumapress SL Separation Solids and Liquors

NREL In-House ☐ Current Subcontractor ☐ CRADA ☐ Other ☒

Name of Project Contact Person	Mark Ruth	Date Samples Delivered	3/21/2000
NREL Notebook	NB# 2275, pp 053-055	Date Work Completed	3/29/2000
Samples Number and Type	10 solids and 22 liquors	Actual Hours Spent	14
Summary of work requested	Analysis of solids for total solids only. Analysis of liquors for total solids content and for free soluble glucose, xylose, and acetic-acid.	Proposed Approach	Analyzed in-house according to NREL Laboratory Analytical Procedures.

Work required: Sample Prep ☒ Acid Digest ☐ HPLC ☒ YSI ☐ GC ☐ CHN ☐ Other: ☐

Results and Comments

Sample		☒ mg/ml G	X	AA		☒ % Dry Weight TS
1 #1 Unwash Hyd.	Avg	nr	nr	nr		54.55
	Std Dev	--	--	--		0.16
	RPD	--	--	--		0.41
2 #1 Unwashed Filt.	avg	14.30	85.46	22.79		12.85
	std dev	0.01	0.08	0.09		0.04
	RPD	0.08	0.13	0.58		0.44
3 Run 1 Test 1 Wash 1 Filt	avg	9.31	55.22	15.00		8.54
	std dev	0.15	0.49	0.01		0.01
	RPD	2.33	1.25	0.10		0.09
4 #2 Cake 1 Wash 117°F	avg	nr	nr	nr		49.68
	std dev	--	--	--		0.28
	RPD	--	--	--		0.79
5 #2 Mother Filtrate	avg	16.42	98.62	23.17		14.65
	std dev	0.03	0.19	0.05		0.07
	RPD	0.24	0.27	0.31		0.64
6 #2 Wash Filtrate	avg	14.39	86.49	21.46		12.87
	std dev	0.00	0.16	0.10		0.01
	RPD	0.04	0.26	0.64		0.07
7 #3 1 Wash 6:1	avg	nr	nr	nr		52.58
	std dev	--	--	--		0.31
	RPD	--	--	--		0.82
8 #3 Mother Filtrate	avg	18.19	109.99	24.93		16.02
	std dev	0.03	0.10	0.17		0.08
	RPD	0.25	0.13	0.98		0.68

#2 Wash Filtrate
#3 1 Wash 6:1
#3 Mother Filtrate

AA=acetic acid; G=glucose; n/a=not applicable; nd=not detected; nr=not requested;TS=total solids; X=xylose

Name(s) of CAT Staff Working on Project:	Reviewed by:
Ray Ruiz	

3

CHEMICAL ANALYSIS & TESTING

Analytical Report

Analysis No.	Page
2000-041 S&L	2 of 3

Results and Comments

Sample		G (mg/ml)	X (mg/ml)	AA (mg/ml)	TS (% Dry Weight)
9 Test #3 Wash Filt	avg	11.85	70.32	16.83	10.75
	std dev	0.02	0.08	0.08	0.00
	RPD	0.25	0.12	0.67	0.03
10 Test #4 1:11:1 Wash	avg	nr	nr	nr	51.08
	std dev	--	--	--	1.24
	RPD	--	--	--	3.42
11 Test #4 Mother Filtrate	avg	18.25	109.89	23.68	16.10
	std dev	0.04	0.11	0.02	0.01
	RPD	0.34	0.14	0.12	0.10
12 Test #4 Wash Filtrate	avg	13.16	78.76	17.69	12.00
	std dev	0.03	0.06	0.10	0.02
	RPD	0.32	0.11	0.80	0.19
13 Hydrol Test #7	avg	nr	nr	nr	65.72
	std dev	--	--	--	0.69
	RPD	--	--	--	1.74
14 Run 1 Test#7 Mother Filt	avg	13.35	78.97	20.89	11.95
	std dev	0.00	0.01	0.05	0.01
	RPD	0.01	0.01	0.31	0.10
15 Run 1 Test 7 Wash 2 Filt	avg	2.59	13.44	3.81	2.21
	std dev	0.03	0.06	0.03	0.00
	RPD	1.66	0.60	0.95	0.16
16 Run 1 Test 7 Wash 3 Filt	avg	0.95	3.62	1.20	0.64
	std dev	0.00	0.00	0.03	0.00
	RPD	0.49	0.05	3.01	0.72
17 7A Run 1	avg	nr	nr	nr	57.09
	std dev	--	--	--	0.82
	RPD	--	--	--	1.52
18 Test 7A Mother Filt	avg	14.40	85.95	21.64	12.86
	std dev	0.02	0.05	0.04	0.06
	RPD	0.21	0.08	0.24	0.68
19 Run 1 Test 7A	avg	2.05	9.60	2.56	1.52
	std dev	0.02	0.05	0.01	0.02
	RPD	1.23	0.72	0.49	1.77
20 Run 1 Test 7A Wash 1 Filt	avg	8.56	49.67	13.17	7.58
	std dev	0.02	0.03	0.05	0.01
	RPD	0.38	0.08	0.54	0.24
21 Test #8	avg	nr	nr	nr	54.70
	std dev	--	--	--	0.08
	RPD	--	--	--	0.21
22 Test #8 Filtrate	avg	14.73	87.97	23.46	13.13
	std dev	0.00	0.08	0.01	0.01
	RPD	0.01	0.13	0.04	0.10

AA=acetic acid; G=glucose; n/a=not applicable; nd=not detected; nr=not requested; TS=total solids; X=xylose

CHEMICAL ANALYSIS & TESTING

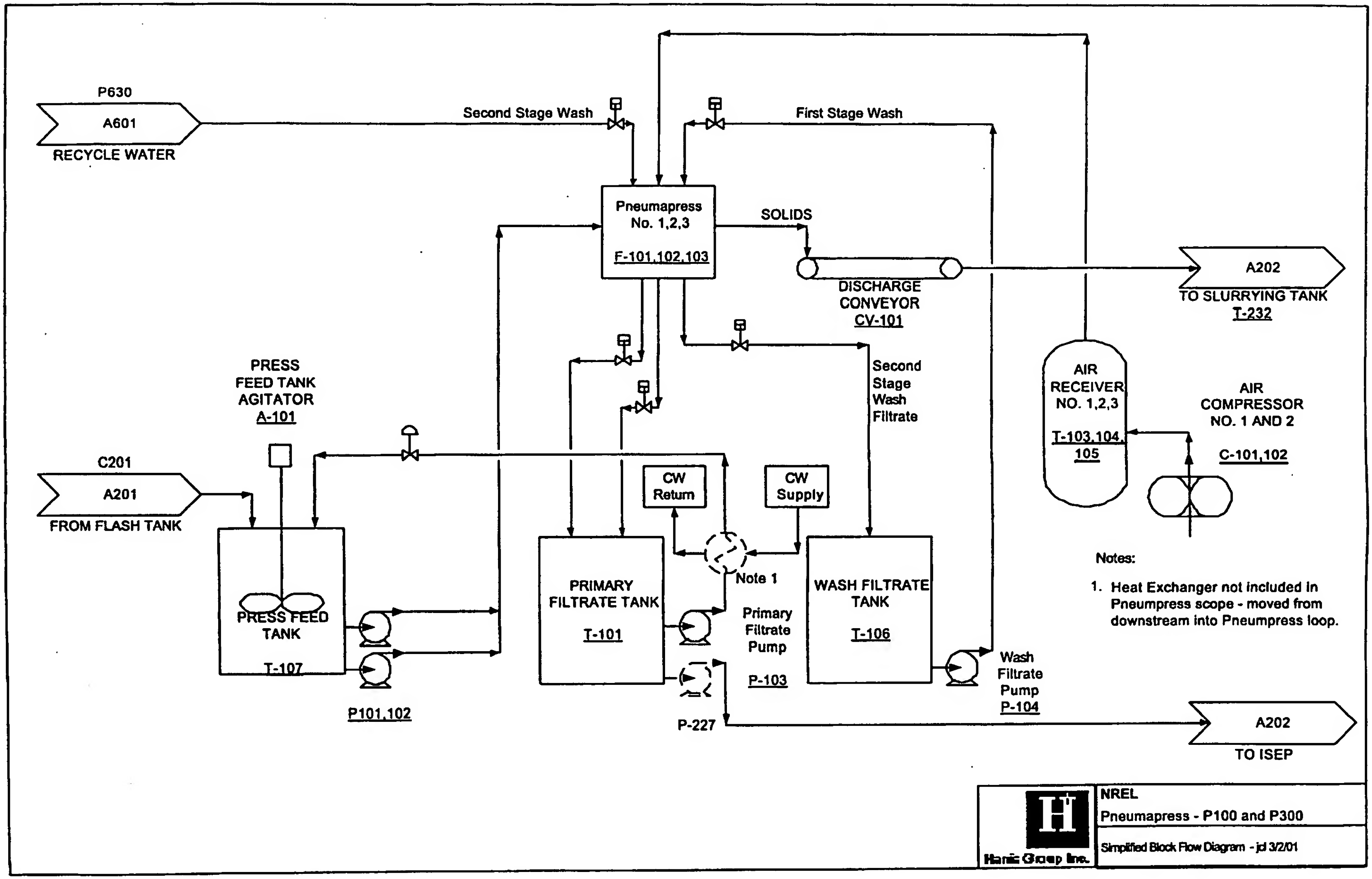
Analytical Report

Analysis #	Page
2000-041 S&L	3 of 3

Results and Comments

Sample		[X] mg/ml G	X	AA	[X] % Dry Weight TS
23 Test #8 Wash 1	avg	8.08	46.18	13.19	7.12
	std dev	0.01	0.02	0.09	0.02
	RPD	0.10	0.05	0.95	0.36
24 Test #8 Wash 2	avg	2.06	9.35	2.93	1.46
	std dev	0.01	0.02	0.02	0.02
	RPD	0.93	0.33	0.89	1.55
25 Test #9	avg	nr	nr	nr	57.46
	std dev	--	--	--	0.44
	RPD	--	--	--	1.08
26 Test #9 Filtrate	avg	13.36	79.31	20.43	11.87
	std dev	0.03	0.08	0.07	0.05
	RPD	0.30	0.10	0.51	0.54
27 Test #9 Wash #1	avg	9.22	53.84	14.64	8.29
	std dev	0.01	0.14	0.02	0.01
	RPD	0.12	0.37	0.22	0.21
28 Test #9 Wash #2	avg	2.87	13.48	4.01	2.13
	std dev	0.02	0.01	0.02	0.00
	RPD	0.81	0.10	0.65	0.18
29 D1 Postdistillate Cake	avg	nr	nr	nr	41.96
	std dev	--	--	--	0.01
	RPD	--	--	--	0.04
30 Test D1 Dist. Filt	avg	nd	1.19	3.61	2.87
	std dev	--	0.00	0.02	0.01
	RPD	--	0.11	0.90	0.62
31 D-2 PostDistillate Cake	avg	nr	nr	nr	88.04
	std dev	--	--	--	0.15
	RPD	--	--	--	0.24
32 Test D-2 Dist Filt	avg	nd	1.40	1.22	2.95
	std dev	--	0.01	0.02	0.01
	RPD	--	1.25	1.89	0.42
	avg				
	std dev				
	RPD				
	avg				
	std dev				
	RPD				
	avg				
	std dev				
	RPD				
	avg				
	std dev				
	RPD				

AA=acetic acid;G=glucose; n/a=not applicable; nd=not detected; nr=not requested; TS=total solids; X=xylose


P630
A601
RECYCLE WATER
Second Stage Wash
First Stage Wash
Pneumapress
No. 1,2,3
F-101,102,103
SOLIDS
DISCHARGE
CONVEYOR
CV-101
A202
TO SLURRYING TANK
T-232
PRESS
FEED TANK
AGITATOR
A-101
C201
A201
FROM FLASH TANK
PRESS FEED
TANK
T-107
P101,102
PRIMARY
FILTRATE TANK
T-101
CW
Return
CW
Supply
Note 1
Primary
Filtrate
Pump
P-103
P-227
Second
Stage
Wash
Filtrate
WASH FILTRATE
TANK
T-106
Wash
Filtrate
Pump
P-104
AIR
RECEIVER
NO. 1,2,3
T-103,104,
105
AIR
COMPRESSOR
NO. 1 AND 2
C-101,102
A202
TO ISEP
Notes:
1. Heat Exchanger not included in Pneumpress scope - moved from downstream into Pneumpress loop.
Harris Group Inc.
NREL
Pneumapress - P100 and P300
Simplified Block Flow Diagram - jd 3/2/01

APPENDIX E
CORROSION DATA

APPENDIX E
CORROSION DATA

C.P. DILLON & ASSOCIATES
Corrosion Control Consultants
1134 Hickory Mills Road
Hurricane, WV 25526
Tel. 304-562-5884
FAX 304-562-6011
CPDILLON@AOL.COM

C.P. Dillon, P.E. *President* | **O.J. Drescher, P.E.** *Vice-President* | **G.B. Elder, P.E.** *Vice-President*

MEMORANDUM

TO: Ms. Andrea Slayton, Harris Group, Seattle, WA

COPY TO: Dr. Julio Moldanado, InterCorr International

SUBJECT: Corrosion Testing Results in NREL Solutions

DATE: January 18, 2000

INTRODUCTION: In accordance with your recent request, I have review the results of the laboratory corrosion tests conducted by Dr. Moldanado relative to the proposed process for conversion of biomasses to ethanol in the presence of dilute sulfuric acid at elevated temperatures. My observations and conclusion are as follows.

OBSERVATIONS: Tests of 24-hours duration were conducted at 190°C (375°F) and 210°C (410°F) in actual NREL solutions of 0.6% and 1.5% sulfuric acid concentration. A solution in which the acid concentration was artificially raised to 2.5% H_2SO_4 was also tested at 80°C (175°F). Following are general corrosion rates in mils per year (mpy) and pit depth in mils in 24 hours.

ALLOYS	2.5% H_2SO_4	1.5% H_2SO_4		0.6% H_2SO_4	
	80°C	210°C		190°C	
	mpy	mpy	Pitting	mpy	Pitting
Alloy 20Cb3	16	800+	3	14	0
Alloy 825	3	400+	3	8	0
Alloy G-30	3	400+	2	7	<1
Alloy C276	6	50	0	15	0
Alloy 2000	4	140	0.5	11	0.5
Zirconium 702	Not run	5	0	6	0

It should be noted that pit depth cannot be extrapolated to an annual rate (as one can with general corrosion) because pits may be arrested as new pits are incurred at other sites. Nevertheless, pitting and crevice corrosion indicate potential problems in the long run.

CONCLUSIONS: In the 80°C liquor at 2.5% H_2SO_4, Alloys 825 and G30 appear to be the obvious choices, with rates of <5 mpy. The latter may cost a little more and probably adds little in the way of improved resistance.

With 0.6% H_2SO_4 at 190°C, alloy 825 (UNS N08825) or Alloy G-30 look to be acceptable (with an adequate corrosion allowance) and would be far less expensive than zirconium (R70200). However, only the alloy 825 is devoid of pitting. The higher rates for Ni-Cr-Mo alloys suggest the presence of organic compounds capable of complexing nickel, such as amines. Also, it should be noted that even a rate of 5-6 mpy for zirconium *might* be unacceptable because of possible hydriding. Although the actual metal loss for zirconium is small, absorption of nascent atomic hydrogen at the local cathodes may cause hydriding, embrittlement and generally unacceptable mechanical properties.

Obviously, none of the superaustenitic alloys are resistant with 1.5% H_2SO_4 at 210°C. Alloy C276 is more resistant to pitting and crevice corrosion under these conditions than are the other nickel-rich alloys but is unacceptable in terms of general corrosion. Zirconium remains a possibility even under these rigorous conditions.

I believe the alloys of choice are Alloy 825 and Alloy G30, subject to actual experience and evaluation.

If we can be of further service, please call on us.

Respectfully submitted.

CP Dillon

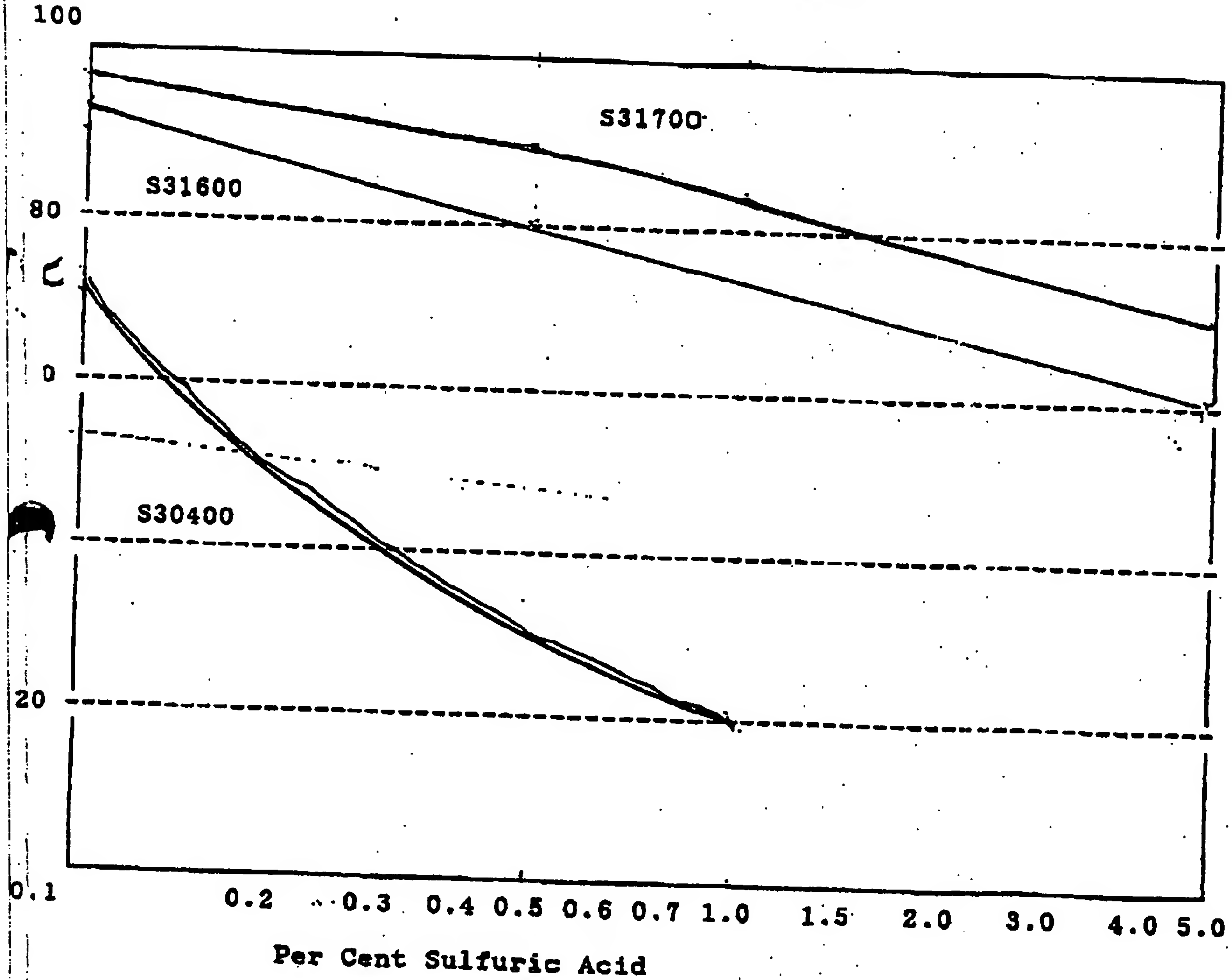
C.P. Dillon

August 1992 Version

Figure 2.1

5 MPY Isolines for 304,316 and 317

Temp. C vs. % Acid



Reference 7

Label Temp

APPENDIX F
REFERENCED REPORTS

Experimental Plan EPD0002
100L SSF of Pretreated Yellow Poplar

Purpose:

This 100L SSF will provide material to the Process Engineering Team for further solid/liquid separation studies.

Procedure:

Fermentor set-up: Batch 160L PDU vessel 445 A with 100 kg of 6% cellulosic solids (~10% w/w total dry solids) and yeast extract/peptone medium and sterilize. At inoculation, add 25 FPU/g cellulose (~2.7 L of enzyme) to the vessel. Inoculate fermentor using the transfer line between the 20L seed vessel and the 160L.

Solids Preparation: An SSF shake flask experiment was conducted comparing unwashed pretreated solids to a 2-volume wash and fully washed pretreated solids. There is indication that a lag in the fermentation occurred in the partially and unwashed solids flasks (analytical analysis is still pending). We are going to go ahead with a wash step. The solids will be slurried in two volumes (one volume of wash is equivalent to an equal weight of water and as is solids) and allowed to settle. The water will be decanted and another two volumes of water will be mixed with the solids, the solids will be allowed to settle and the water will be decanted. The water volumes will be tracked so that the amount of water left with the solids will be known. The slurry will be added to the fermentor and sterilized.

Strain/Inoculum Procedure: *Saccharomyces cerevisiae* D_5A. Two mL of frozen culture will be inoculated into 100 mL of YPD medium, incubated overnight (~16 hours), transferred to 1L of YPD and incubated for about 12 hours then transferred to 10L of YPD. Again, the culture will be incubated for 12 hours then transferred to the 100L SSF vessel.

Media: 6% w/w cellulosic solids (~10% total dry solids)
0.5% w/w yeast extract
1.0% w/w peptone
100L working volume

Fermentation Conditions: Temperature 37°C
pH 5.3 controlled with 2N NaOH
Agitation 100-150 rpm depending on mixing
10% v/v inoculum transfer (about and O.D. @ 600 nm of 0.5)
25 FPU/g cellulose of commercial enzyme (CPN 55 FPU/mL)

Analytical: Ethanol measurement by GC, glucose by YSI, solids analysis of solid residue before and after SSF is completed.

Post SSF: The vessel will be pasteurized at 90°C for 30-45 minutes to deactivate the enzyme and yeast. This will also reduce the ethanol in the broth because of evaporation.

ACID HYDROLYSIS SUPPORT
FIRST AND SECOND STAGE HYDROLYSIS TESTING

MPO NO. DCO-8-18081-0

April 14, 2000

Submitted by
Millicent Moore, Technical Monitor
Tennessee Valley Authority
Public Power Institute
Muscle Shoals, AL 35662

Acid Hydrolysis Support
Summary Report

Introduction

TVA received approximately 5000 lb. of ¾ inch, wet, pulp-size aspen hardwood chips from NREL contained in boxes ranging from 800-1,200 lb. each. Using these wood chips, TVA conducted two tasks for NREL. First, the wood chips were used to produce 650 gallons of hydrolyzate slurry using low concentration acid hydrolysis in TVA's biomass pilot plant. Before this could be done, the equipment had to be modified to bypass the screw press. The unpressed slurry was then collected in 55 gallon drums, some of which were shipped to NREL for testing. A sample of each drum was kept by TVA for laboratory analysis.

The second task conducted by TVA was second stage hydrolysis in a lab-scale reactor. The pretreated solids from the first stage hydrolysis were used to produce about 4 liters of hydrolyzate. A small sample of the liquid was kept by TVA for analysis, and the remaining liquid was then shipped to NREL for testing. The results of the two tasks will be summarized in this report.

Task 1—Production of First Stage Pretreated Biomass

The reaction vessel used in the first test was a zirconium-lined digester manufactured by Sunds Defibrator, Inc. The continuous reactor is equipped with a compactor screw at the inlet where the feedstock was fed to the agitated slurry. After the set retention time, the slurry was then released to a receiving vessel before being stored in the drums.

Wood chips were tested in the laboratory for moisture content. It was determined that the chips had a moisture content of 44%. The wood chips were fed to the hydrolyzer at a rate of 200 lb/hr which correlates to a feed rate of 112 lb/hr of dry wood and 88 lb/hr of water. In addition, a dilute acid solution was fed at a rate of 260 lb/hr and steam was fed at a rate of 100 lb/hr. The feed rates chosen gave a liquid/solids ratio of 4:1 and an acid concentration of 0.55% in the liquid phase. Both the liquid/solids ratio and the acid concentration values were requested by NREL. The test was conducted at a temperature of 343°F and a pressure of 112 psig. A retention time of 15 minutes was chosen for the test.

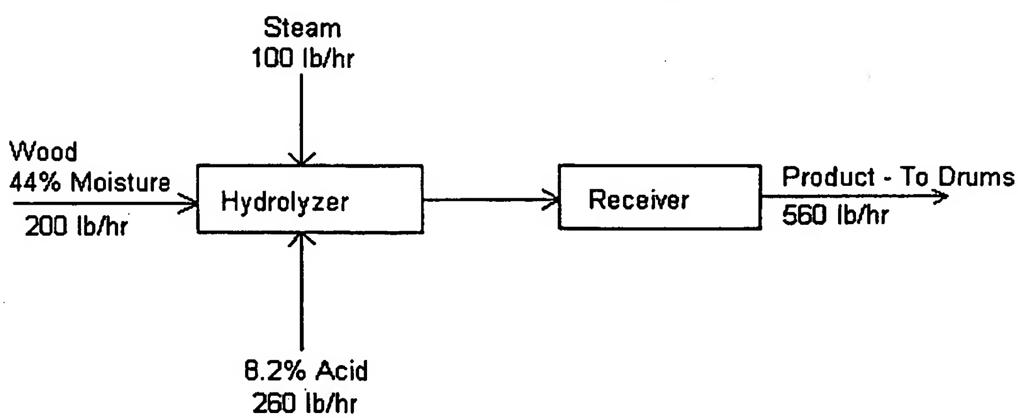
Pilot Plant Performance

The pilot plant was operated for two days for a total of approximately 18 hours. On the first day, the plant ran for approximately 6 hours. During this time, plant shakedown was conducted, ensuring that the run would go smoothly. The shakedown included testing the operation of valves and screws, removal of the screw press, and the installation of a chute used to fill the drums. On the second day, the plant operated satisfactorily. The requested 650 gallons of slurry was produced.

Material Balances and Flow Diagram

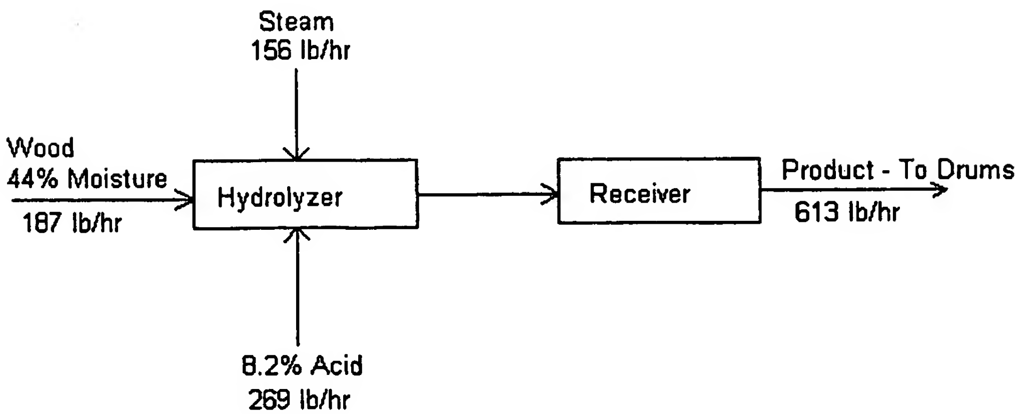
Preliminary material balance calculations were performed based on a moisture content of 44% in the wood chips, a maximum wood chip feed rate of 200 lb/hr, and a steam feed of 100 lb/hr. From these calculations, it was determined that an acid flow of 260 lb/hr would give the desired 4:1 liquid-to-solids ratio. Below, Figure 1 gives a graphical representation of these flows.

Figure 1. Theoretical Material Flows and Flow Diagram



Readings of the process variables were taken every thirty minutes during plant operation. Time-weighted averages of these variables are shown in Figure 2.

Figure 2. Actual Average Material Flows and Flow Diagram



Preliminary Results

The liquid from the samples collected from four drums (11, 13, 14, and 16) during the first stage hydrolysis conducted in the pilot plant was separated from the solids and analyzed in the laboratory for sugar content, acetic acid, HMF, and furfural. In addition, the moisture content was analyzed for drums 11 and 16. There were three moisture tests for each drum, the average moisture content for drums 11 and 16 was 59.7% and 59.3%, respectively. The results of the analysis are given in below in Table 1.

Table 1. Composition of First Stage Hydrolyzate from Pilot Plant in mg/mL.

Drum	Glucose	Xylose	Galact.	Arab.	Mann.	Acetic Acid	HMF	Furfural
11	9.90	76.20	<1.25	2.25	11.70	21.10	0.29	1.60
13	11.60	62.90	<1.25	1.90	8.40	18.00	0.36	1.78
14	11.60	80.00	-	1.90	9.33	19.80	0.31	1.74
16	10.70	82.00	<1.25	2.25	11.70	21.20	0.31	1.60

Task 2—Production of Second Stage Pretreated Biomass

Second stage hydrolysis was conducted in the laboratory on a sample of the first stage hydrolyzate slurry from the pilot plant. The reactor used in this process was a 2-gallon Parr batch reactor. Using a retention time of 2 minutes, the material was continuously stirred at 210°C.

Preliminary Results

The liquid from the second stage hydrolysis process was separated from the solids and analyzed in the laboratory for sugar content, acetic acid, HMF, and furfural. The results of the analysis are given below in Table 2. The first set of data, labeled Sample A, was the original composition of the sample of first stage solids from the pilot plant before second stage hydrolysis was conducted. This was included for comparison with the second stage compositions. Runs B, C, and D show the compositions of the hydrolyzate samples after second stage hydrolysis was conducted.

Table 2. Composition of Second Stage Hydrolyzate from Lab in mg/mL.

Sample	Glucose	Xylose	Galact.	Arab.	Mann.	Acetic Acid	HMF	Furfural
A	11.10	74.40	3.75	2.11	1.02	20.30	0.26	1.52
B	57.37	<0.50	<0.50	<0.50	<0.50	1.97	6.03	2.01
C	55.87	<0.50	<0.50	<0.50	<0.50	1.96	6.23	6.25
D	54.30	<0.50	<0.50	<0.50	<0.50	2.28	6.02	2.02

Conclusions

First stage hydrolysis of the wood chips resulted in an average xylose concentration of 75.2 mg/mL. Though the raw chips were not analyzed for hemicellulose content, the relatively high xylose concentration suggests a high conversion efficiency for the first-stage hydrolysis step. Furfural concentrations were at low levels (1.6-1.78 mg/mL) which indicates a small amount of product degradation.

Second-stage lab hydrolysis conditions resulted in somewhat lower concentrations of glucose in the hydrolyzate stream. HMF concentrations were high indicating significant degradation of the glucose. It is recommended that subsequent second stage hydrolysis tests be conducted using slightly milder conditions.